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# Evaluation of Hazardous Waste Incineration in a Lime Kiln Rockwell Lime Company

Monsanto Research Corp., Dayton, OH

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# EVALUATION OF HAZARDOUS WASTE INCINERATION IN A LIME KILN: ROCKWELL LIME COMPANY

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Results show average DRE's greater than 99.99 percent for each POHC and little change in pollutant emissions from baseline to waste fuel test conditions. In addition, material balance results show that 95 percent of chlorine enters the process from the limestone feed and the chlorine exits the kiln in the baghouse dust and lime product at 61 percent and 38 percent, respectively.

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# FOREWORD

Industrial hazardous wastes represent a threat to our environment and our health. Many of these wastes are known to contain organic substances with documented and suspected carcinogenic and toxic properties. Industrial treatment and disposal practices of these wastes are regulated by the Office of Solid Waste of the U.S. Environmental Protection Agency under the Resource Conservation and Recovery Act (RCRA). The Industrial Environmental Research Laboratory (IERL-Cincinnati) of the Office of Research and Development assists in developing and demonstrating new methodologies for the safe treatment and disposal of these wastes.

In 1981 the Office of Solid Waste, under RCRA, developed regulations for the incineration of hazardous waste. Industrial boilers and kilns are exempt from these regulations. In response to environmental concerns regarding this practice, the Agency has undertaken a multidisciplinary research program to determine the operating practices and destruction efficiency of boilers and kilns burning hazardous wastes as information necessary to regulation development and the Regulatory Impact Analysis (RIA) required under Executive Order 12291.

One important area of this research program is the field testing of full-scale industrial kilns burning hazardous waste for disposal and energy recovery. This report documents the results of a field test program to characterize destruction efficiencies of representative lime kilns burning hazardous wastes under typical industrial operating conditions.

# ABSTRACT

During a one-week test burn, hazardous waste was used as supplemental fuel and co-fired with petroleum coke in a lime kiln in eastern Wisconsin. Detailed sampling and analysis was conducted on the stack gas for principal organic hazardous constituents (POHCs), particulates, particulate metals, HCl, $SO_2$, $NO_x$, CO, and THC and on process streams for metals and chlorine. POHCs were also analyzed in the waste fuel. Sampling was conducted during three baseline and five waste fuel test burn days. The program objectives were to determined the destruction and removal efficiency (DRE) for each POHC, determine concentration of stack gas pollutants under baseline and waste fuel test burn conditions, determine the fate of chlorine, sulfur, and trace metals in the kiln process, and evaluate kiln performance when operating with hazardous waste as supplemental fuel.

Results show average DRE's greater than 99.99% for each POHC and little change in pollutant emissions from baseline to waste fuel test conditions. In addition, material balance results show that 95% of chlorine enters the process from the limestone feed and the chlorine exits the kiln in the baghouse dust and lime product at 61% and 38%, respectively.

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## SECTION 1

## INTRODUCTION

Cofiring of hazardous wastes in high temperature industrial processes is an attractive alternative to hazardous waste incineration. The alternative makes use of the waste's heat content. Many processes including cement and dolomite kilns, glass furnaces, steel furnaces, and some industrial boilers operate at temperatures and residence times similar to those required for incinerators dedicated to incineration of hazardous wastes and thus offer opportunities for cofiring. In addition to the savings derived from the heat value in the waste fired, the use of existing industrial equipment provides an environmentally acceptable alternative to conventional hazardous waste disposal while not requiring the capital expenditure that would otherwise be required if a separate incinerator to process and incinerate a given amount of hazardous waste is to be built.

Lime kilns, because of their high energy use, are an excellent example of this concept. Such kilns typically operate at temperatures over 1,093°C (2,000°F), have gas residence times in excess of 1.5 seconds, and have a highly turbulent combustion zone. However, the need exists for data that shows the effect of hazardous waste cofiring on the emissions from the lime process.

The State of Wisconsin Department of Natural Resources (DNR) and EPA Region V, in implementing RCRA regulations, issued a temporary permit to Rockwell Lime Company to conduct a hazardous waste test burn. This test would allow the burning of hazardous liquid waste as supplemental fuel along with petroleum coke. If proven feasible to use, the waste fuel would replace natural gas as a fuel component and offset the fuel costs by using a less expensive fuel.

Coordinating activities with the Wisconsin DNR and EPA Region V, the U.S. Environmental Protection Agency Office of Research and Development (EPA-IERL) in Cincinnati, Ohio contracted Monsanto Company (MC) to perform sampling and analysis of stack gases and process samples during the test burn.

The primary objectives of the sampling and analytical program were to (1) determine the effects of cofiring petroleum coke and liquid organic hazardous waste on the emissions from the kiln, (2) determine the fate of the principal organic hazardous constituents (POHCs) and determine destruction and removal effeciencies (DRE), (3) determine the fate of chlorine and trace metals from the waste fuel, (4) determine the concentration of sulfur dioxide ($SO_2$), nitrogen oxides ($NO_x$), particulates, hydrogen chloride (HCl), metals, total hydrocarbons (THC), and carbon monoxide (CO) in the stack gas at baseline and waste fuel test burn conditions, and (5) evaluate kiln operation during hazardous waste fuel burning condition. The testing would assist Rockwell Lime Company to determine if they can operate while using a new fuel and should apply for a permit, and will provide to the Wisconsin DNR and the EPA Region V the data necessary to determine whether a permit can be issued to Rockwell Lime Company to burn hazardous waste. The testing also will provide the EPA-IERL with additional data in their research on the incineration of hazardous waste and the environmental problems associated with incineration.

The sampling and analysis program was conducted at the Rockwell Lime Company in Rockwood, Wisconsin on April 15 and April 29 through May 6, 1983 under baseline (no waste fuel burned) and waste fuel conditions (coke and waste fuel burned). The program included (1) the sampling and analysis of stack gas for particulates, HCl,

$SO_2$, $NO_x$, CO, THC, $O_2$, metals, and POHCs; and (2) the collection of process samples, including waste fuel, limestone feed, lime product, coke, and baghouse dust, and their analysis for a variety of components.

The following sections describe the site facilities and process (Section 2), the sampling and analysis program (Section 3), the results of sampling and analysis (Section 4), the evaluation of the results (Section 5), the test conclusions (Section 6), and the quality control results (Section 7).

# SECTION 2

# DESCRIPTION OF PROCESS AND WASTE FUEL SYSTEM

The Rockwell Lime Company operates a lime kiln in Rockwood, Wisconsin, which is located approximately 10 miles north of Manitowac in eastern Wisconsin. Production of lime, which is converted into various forms such as hydrated and barn lime, is approximately $1.3 \times 10^6$ kg (1,430 tons) per week and varies with product demand.

## 2.1 PROCESS DESCRIPTION

Plant facilities consist of (1) quarries from which raw limestone material is extracted, separared by size, trucked to the storage area and fed to the limestone feed hopper as needed for lime production; (2) two rotary kilns (of which only one was in operation during testing) which convert the limestone feed into the lime product; (3) lime product handling and storage silos; (4) a coal rail and feed system; (5) a natural gas feed system; (6) a kiln burner system for coal and natural gas; and (7) a kiln emissions handling system including radiator exhaust gas coolers, baghouse, fan, stack, and baghouse dust silo. Temporary facilities located on site included MRC sampling vehicles, a waste fuel storage tanker, and a waste fuel pumping and burner system. Figure 2-1 illustrates the kiln plant layout including major equipment and the waste storage area. Figure 2-2 provides a view of kiln No. 2 from the burner end. Figure 2-3 shows the limestone quarry as viewed from the stack.

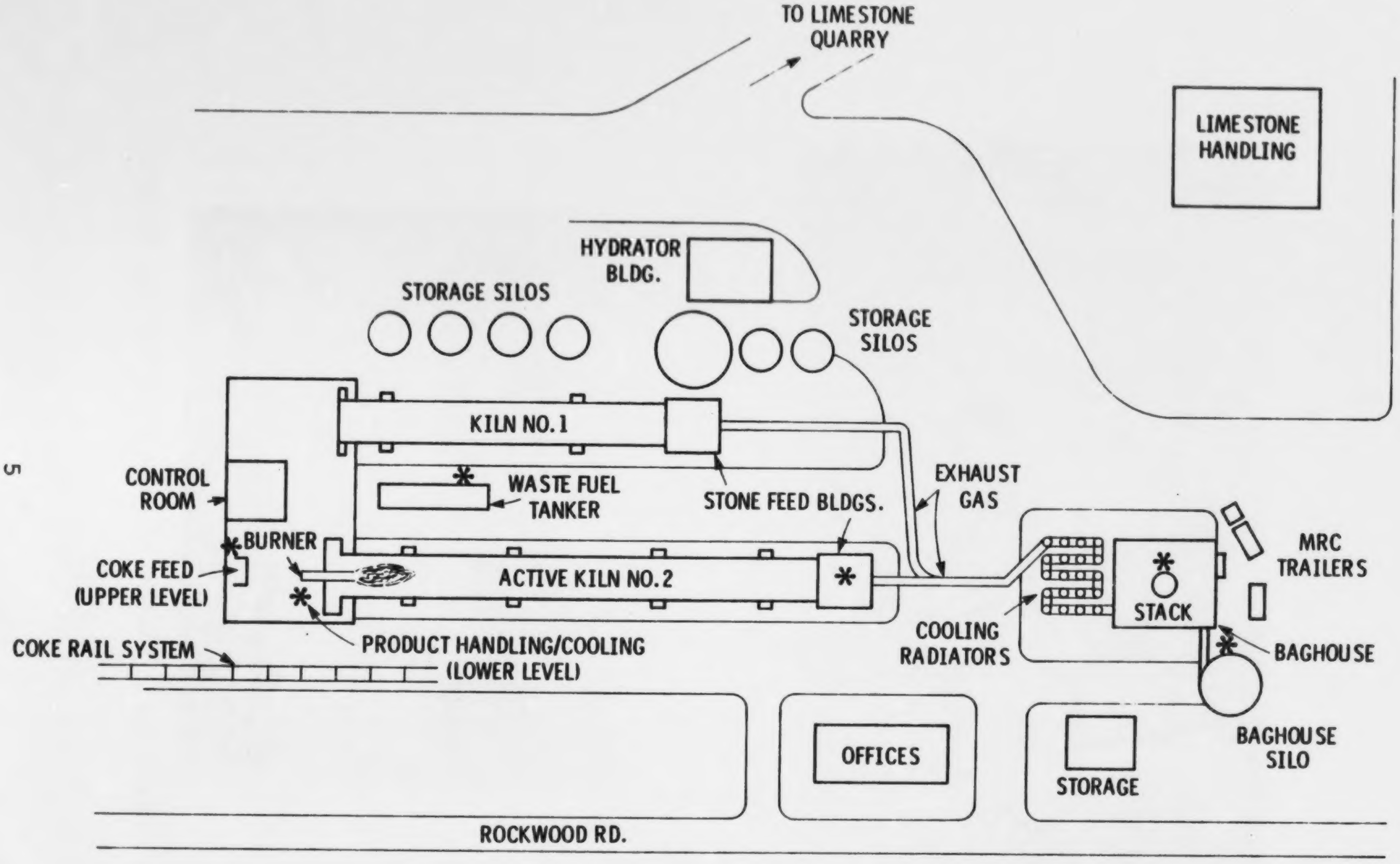

TO LIMESTONE QUARRY
LIMESTONE HANDLING
HYDRATOR BLDG.
STORAGE SILOS
STORAGE SILOS
KILN NO. 1
CONTROL ROOM
WASTE FUEL TANKER
STONE FEED BLDGS.
EXHAUST GAS
BURNER
COKE FEED (UPPER LEVEL)
ACTIVE KILN NO. 2
MRC TRAILERS
STACK
COOLING RADIATORS
BAGHOUSE
COKE RAIL SYSTEM
PRODUCT HANDLING/COOLING (LOWER LEVEL)
BAGHOUSE SILO
OFFICES
STORAGE
ROCKWOOD RD.

Figure 2-1. Plan view of Rockwell Lime site in Rockwood, Wisconsin (not to scale). Sample locations shown by asterisk.

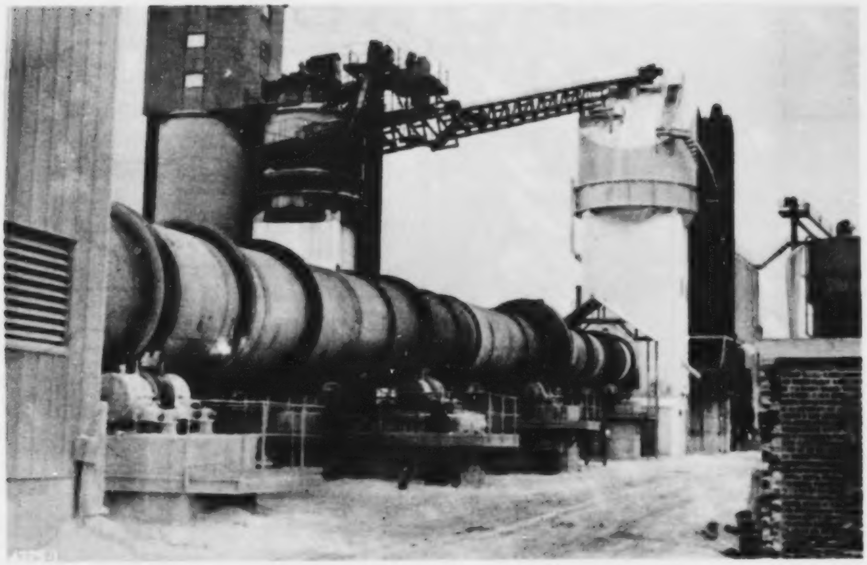


Figure 2-2. Pictorial view of kiln No. 2.

Figure 2-3. Pictorial view of limestone quarry.

The process, shown schematically in Figure 2-4, involves feeding the limestone into a rotary kiln and heating the limestone to a temperature of 1,093°C (2,000°F). The calcining which is achieved by interfacing hot gases with limestone, drives off $CO_2$ from the limestone, leaving the lime product (CaO). The kiln, with refractory linings, is 2.4 m (8 ft) in diameter and 67.1 m (220 ft) long. The kiln rotates slowly (approximately one revolution per minute) and has a gentle slope (6 cm/m of length) to allow material to pass through by gravity. The kiln also has a countercurrent flow pattern, i.e., solid materials travel in one direction and hot gases and dust emissions travel in the opposite direction. Limestone is fed into the kiln at the upper end at a rate of approximately 15,440 kg/hr (34,000 lb/hr). At the opposite end of the kiln, a mixture of coal and natural gas is burned at the rates of approximatly 1,450 kg/hr (3,200 lb/hr) and 142 $m^3$/hr (5,000 $ft^3$/hr), respectively, to provide a heat input of approximately 14,700 kw (50 million Btu/hr) or approximately 6.5 million Btu/ton of lime product. During the waste fuel test burn, waste fuel was substituted for the natural gas and fed to the kiln at approximately 2.3 to 11 L/min (0.6 gal/min to 2.9 gal/min). As the limestone feed travels down the inclined rotating kiln, it passes through various temperature zones, and the hot gases calcine the limestone into the lime product. The product is produced at a rate of approximately 7,720 kg/hr (17,000 lb/hr). After heating and transformation in the kiln, the lime product is cooled by air and either stored in silos or hydrated prior to storage in silos where it is held until sold.

In addition to observing the lime product as it exits the kiln to maintain high product quality, the kiln operators also observe air flow rates and temperatures, limestone feed rates, coke and natural gas feed rates, kiln rpm and temperature, baghouse pressures, storage silo capacities, and the percent oxygen and percent combustibles of the kiln exhaust gases.

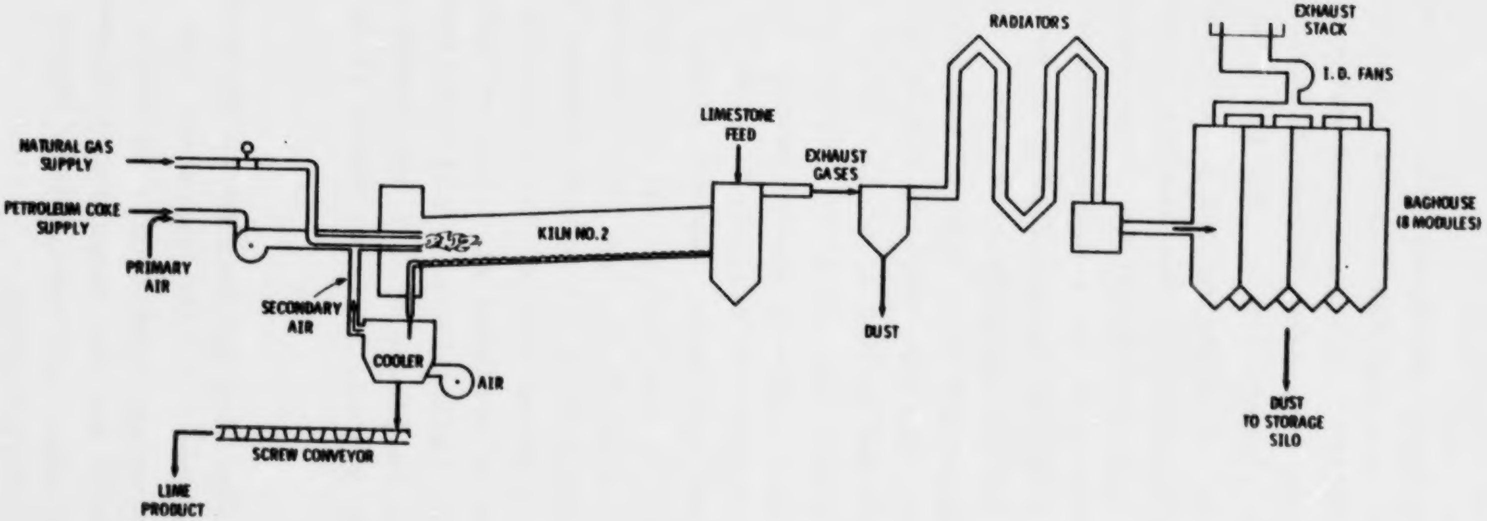

NATURAL GAS SUPPLY
PETROLEUM COKE SUPPLY
PRIMARY AIR
SECONDARY AIR
COOLER
AIR
SCREW CONVEYOR
LIME PRODUCT
KILN NO. 2
LIMESTONE FEED
EXHAUST GASES
DUST
RADIATORS
EXHAUST STACK
I. D. FANS
BAGHOUSE (8 MODULES)
DUST TO STORAGE SILO

Figure 2-4. Schematic diagram of lime kiln process.

The kiln is fed by primary air mixed with the gas and coke and secondary (heated) air from the lime product cooler, which provides oxygen for the combustion of the coke and natural gas (or waste fuel). The kiln exhaust gases pass through a series of large radiator coolers to cool the gases prior to their entering the baghouse, which removes particulates and $SO_2$ from the gas stream. The gases then pass through the induced draft fans and out the stack at approximately 200°C (392°F) and 5.5 m/s (18 ft/s). Figure 2-5 provides a view of the baghouse, stack, and cooling radiators. Except for process samples, all sampling was conducted on emissions from the stack. Sampling locations are marked with an asterisk (*) in Figure 2-1.

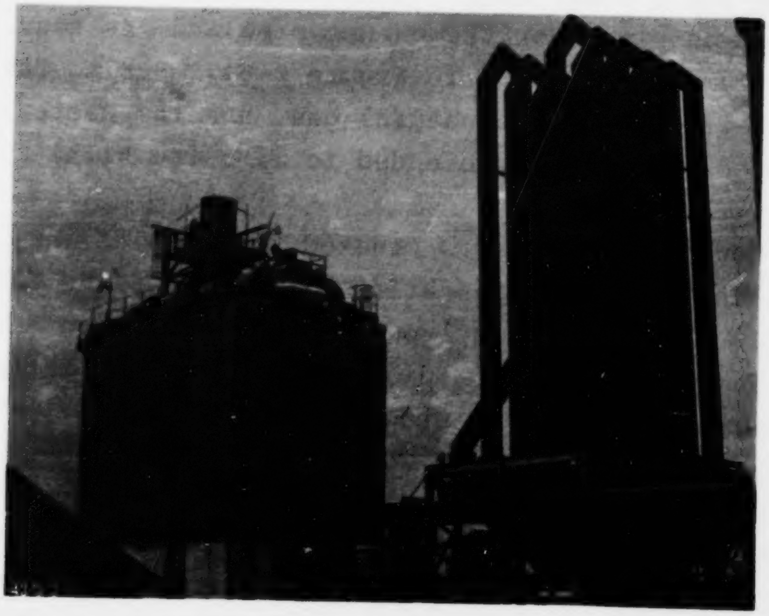


Figure 2-5. Baghouse, stack and cooling radiators.

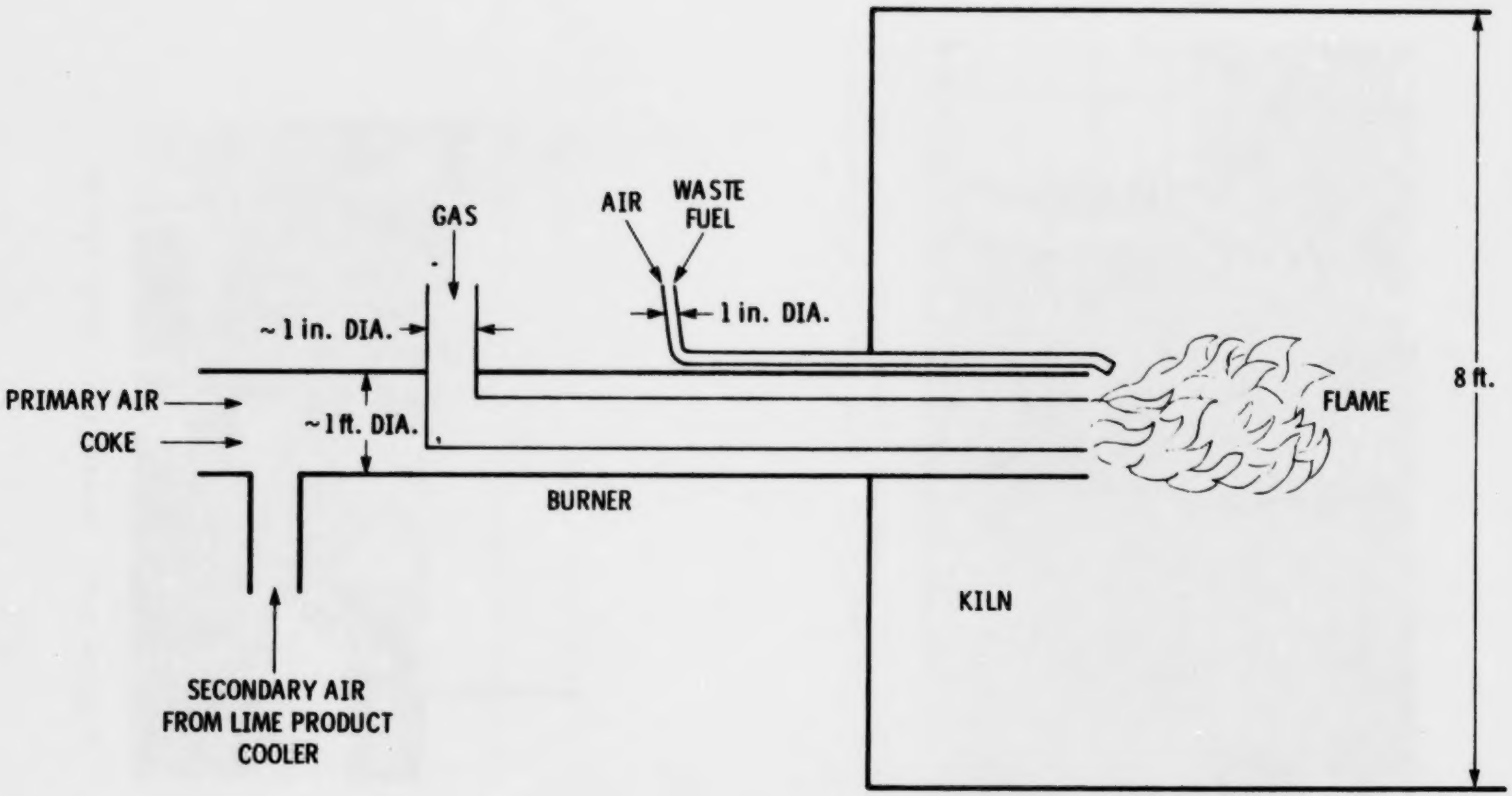
Particulates and $SO_2$ are removed in the eight-module baghouse that was installed in 1980. The bags are cleaned by reverse air sequentially once every two hours, and the collected dust is stored in a silo and mixed with water to granulate. Some of the dust is sold and the remainder is disposed of in the quarry. No dust is reinjected into the kiln.

## 2.2 WASTE FUEL SYSTEM

During baseline runs, a blended combination of petroleum coke and natural gas was used to fire the kiln. As shown in Figure 2-6, the gas was fed to the kiln through the center tube, and the coke was fed through the outer burner tube. Figure 2-7 provides a view of the burner end of the kiln.

During the waste fuel runs, a temporary 1-inch diameter stainless steel pipe was placed on the burner pipe and its nozzle pointed into the flame (also shown in Figure 2-6). During the waste fuel runs, waste fuel replaced natural gas, and the waste fuel and petroleum coke were fed unblended to fire the kiln.

The hazardous waste fuel was trucked to the site and stored in a 5,000-gallon tanker between kilns 1 and 2 near the burner end (see Figure 2-1). The waste fuel pump, located next to the tanker (as shown in Figure 2-8), was a diaphragm-type pump that pumped waste fuel through the stainless steel pipe to the flame. Air was added to the pipe to supply oxygen for combustion and to cool the stainless steel pipe. An additional pump also was used to recirculate the waste fuel in the tanker. Flow rate measurement of waste fuel was obtained using a sonic flow meter attached to the waste fuel pipe and by sticking the tanker truck every hour and calculating a change in depth.



GAS
AIR
WASTE FUEL
~ 1 in. DIA.
1 in. DIA.
PRIMARY AIR
COKE
~ 1 ft. DIA.
BURNER
FLAME
8 ft.
KILN
SECONDARY AIR FROM LIME PRODUCT COOLER

Figure 2-6. Burner configuration.

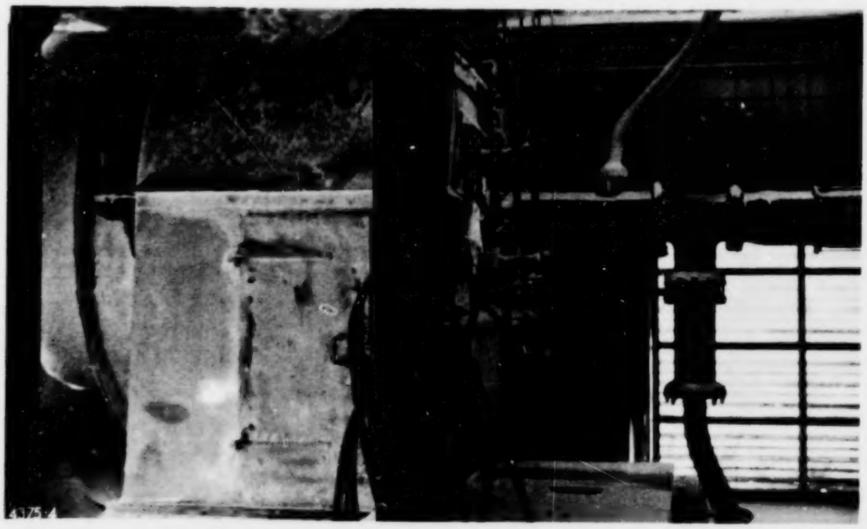


Figure 2-7. Kiln burner area, coke, air, and gas feed pipe, and wastefuel feed pipe.

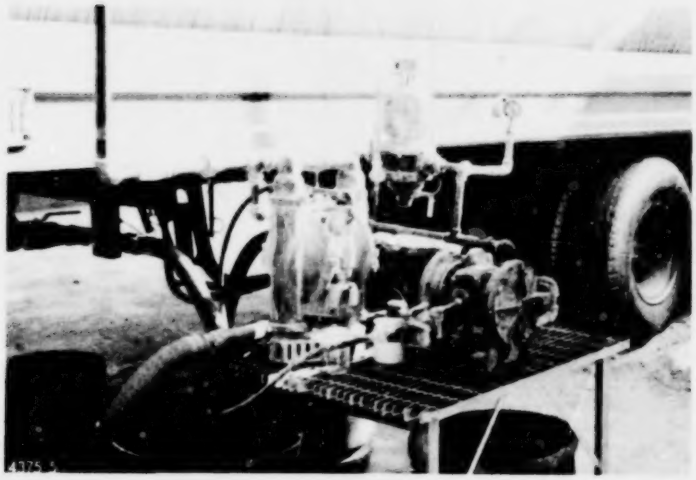


Figure 2-8. Waste fuel pump system.

The temporary permit issued by the Wisconsin DNR allowed a maximum of 5,000 gallons to be burned during waste fuel testing. The waste fuel was provided by Hydrite Chemical Company and consisted primarily of solvents from lacquer thinners, alcohols, still bottoms, paint wastes, and a small fraction of chlorinated hydrocarbons (0.4%). A more detailed analysis of the waste fuel was conducted as part of the analytical program. Additional chlorinated organics (tetrachloroethylene and trichloroethylene) were spiked to the waste fuel prior to the test to bring the total chlorine content to approximately 3.0%, which would allow easier evaluation of the destruction of the chlorinated species in the waste fuel. During the waste fuel test conditions, the waste fuel ranged from 8% to 36% of the Btu input to the kiln, and petroleum coke ranged from 64% to 90%. Comparatively, under baseline conditions, the petroleum coke averaged 90% and the natural gas averaged 10% of the Btu input to the kiln.

## 2.3 KILN OPERATION AND FLAME CONTROL

For the thermal decomposition of limestone, three essential factors must be considered [1]:

- A desired temperature is required (for dissociation of carbonates from limestone)
- This temperature must be maintained for a certain duration
- The generated $CO_2$ gas must be rapidly removed

The required temperature and duration is a function of the limestone feed size, calcium and dolomite contents and impurities in the limestone, kiln size, etc., with the objective of producing a completely calcined but soft-burned lime. The kiln temperature and duration are based primarily on the kiln flame, and fuel and oxygen level used. The optimum flame is difficult to achieve, but such a flame will maximize the higher Btu reaction of $CO_2$ and provide good quality lime product. In terms of hazardous waste incineration, the optimum flame also will provide excellent conditions for high destruction of organics in the waste.

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[1] Kirk-Othmer Encyclopedia of Chemical Technology. 2nd edition. Vol. 12. John Wiley and Sons, Inc. New York, NY, 1967. pp. 434-437.

The usual products of complete combustion are $CO_2$, $H_2O$, $SO_x$, and $NO_x$, with CO present at times of incomplete combustion. Within the kiln industry, recording instruments that track the amount of CO and $O_2$ in the exhaust gas have been used to control the kiln flame. The kiln operator attempts to keep the $O_2$ content as low as possible while maintaining the CO content near zero. It was assumed that this operating procedure assured sufficient $O_2$ to convert all CO to $CO_2$ for complete combustion while keeping the heated waste surplus air to a minimum. However, it has been proven that sections of the flame within the kiln can suffer from insufficient $O_2$ (localized reducing conditions) even though the above described burning restrictions were met. The resulting flame required more time to complete its combustion reactions and became longer and less intense (the highest possible flame temperature was not realized). These areas of reducing conditions in the flame were difficult to eliminate because operator "eye-ball" adjustment was the only option available.

Recently, on-stream $NO_x/SO_x$ analysis has eliminated (or made elimination possible) the localized reducing conditions. On-stream $NO_x/SO_x$ analyzers can record the actual amount of nitrogen-sulfur compounds being created by the kiln flame. Minor kiln adjustments can cause large fluctuations in the production of these compounds. Attempts to use $CO_2$ measurements for the same purpose met with failure because the $CO_2$ contribution from calcining lime tended to vary wildly from minute to minute. The flame that maximized the $NO_x$ and minimized the $SO_x$ in the exhaust gas stream was the most economical flame that produced the best product and lowered the amount of waste dust. This is the hottest or most intense flame possible within the kiln system. Similarly, this high intensity flame most likely provides the best conditions for high destruction of organics contained in a supplemental hazardous waste fuel such as the fuel used in this test burn [2].

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[2] Communications with Jack Mander, Kilns and Fuels Technology, North Hampton, PA.

## SECTION 3

## SAMPLING AND ANALYSIS PROGRAM AND TEST MATRIX

The sampling and analytical program was designed to (1) identify the major pollutants from burning waste fuel in a lime kiln, (2) quantify their respective emission rates, (3) determine the destruction and removal efficiency (DRE) of the POHCs, (4) provide information for a mass balance around the process for metals and chlorine, (5) determine the fate of metals and chlorine from the waste fuel, and (6) determine the use of sulfur dioxide ($SO_2$), $NO_x$, and/or carbon monoxide (CO) to evaluate kiln performance and DREs for the POHCs. Table 3-1 summarizes the overall test program and lists each sampling and analytical method used. Pollutants which were sampled in the stack gas include POHCs (tetrachloroethylene, trichloroethylene, methylene chloride, 1,1,1-trichloroethane, methyl ethyl ketone, and toluene), particulate matter, particulate trace metals, hydrogen chloride, sulfur dioxide, nitrogen oxides, carbon monoxide, total hydrocarbons, and oxygen. In addition, the distribution of metals and chlorine were measured in all of the process input and output streams (i.e., the coke feed, waste fuel feed, limestone feed, lime product, and baghouse dust). Waste fuel, coke, baghouse dust, and lime product samples were submitted for sulfur analyses. Coke samples were analyzed for ash and Btu content. Waste fuel samples also were analyzed for POHCs and Btu content.

Sampling was conducted under baseline conditions (i.e., no waste fuel burned) on April 15, 29, and 30. Sampling at waste fuel conditions (i.e., waste fuel burned) was conducted from May 2 to May 6. Sampling was originally scheduled to be continuous from

## TABLE 3-1. SUMMARY OF ROCKWELL LIME KILN SAMPLING AND ANALYTICAL PROGRAM

| Parameter | Sampling method | Analytical method |
|---|---|---|
| STACK GAS | | |
| 1. POHCs[a] | Volatile organic sampling train (VOST) | GC/MS, thermal desorption and SIM |
| 2. Particulate matter | EPA Method 5 | EPA Method 5 |
| Metals on particulate | EPA Method 5 | ICAP |
| Dioxins on particulate | EPA Method 5 | GC/MS |
| 3. Hydrogen chloride | Impinger absorption in 0.5 M NaoAc (back half of EPA Method 5) | Specific ion electrode |
| 4. Carbon dioxide and oxygen | EPA Method 3 | Fyrite |
| 5. Nitrogen oxides | Continuous | Chemiluminescence photometric analyzer |
| 6. Sulfur dioxide | Continuous | Pulsed fluorescence TECO analyzer |
| 7. Carbon monoxide | Continuous | Infrared-EPA Method 10 |
| 8. Total hydrocarbons | Continuous | Flame ionization detector |
| 9. Oxygen | Continuous | Teledyne's micro-fuel cell |
| 10. Waste fuel | | |
| POHCs | Grab → composite | GC/MS |
| Metals | Grab → composite | ICAP |
| Chlorine, sulfur | Grab → composite | ASTM D240-64 |
| Btu content | Grab → composite | ASTM D482-IP4 |
| 11. Baghouse dust | | |
| Metals | Grab → composite | ICAP |
| Chlorine, sulfur | Grab → composite | XRF |
| Dioxins | Grab → composite | GC/MS |
| 12. Lime product | | |
| Metals | Grab → composite | ICAP |
| Chlorine, sulfur | Grab → composite | XRF |
| 13. Dry limestone feed | | |
| Metals | Grab → composite | ICAP |
| Chlorine | Grab → composite | XRF |

(continued)

TABLE 3-1 (continued)

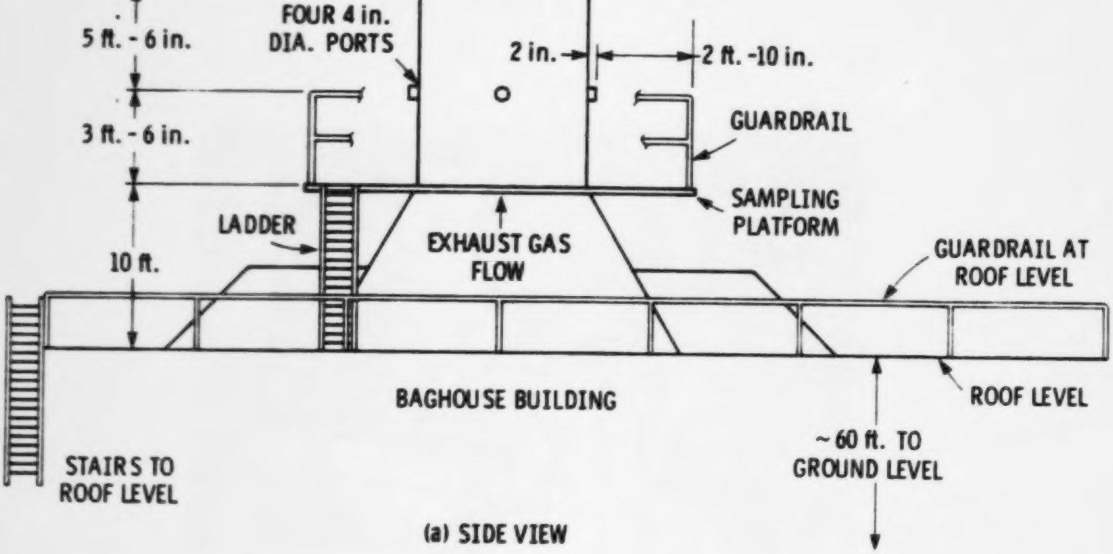
| Parameter | Sampling method | Analytical method |
|---|---|---|
| 14. Primary fuel coke | | |
| Metals | Grab → composite | ICAP |
| Chlorine, sulfur | Grab → composite | XRF |
| Btu content | Grab → composite | ASTM D240-64 |

[a] Tetrachloroethylene, trichloroethylene, methylene chloride, 1,1,1-trichloroethane, methyl ethyl ketone, and toluene.

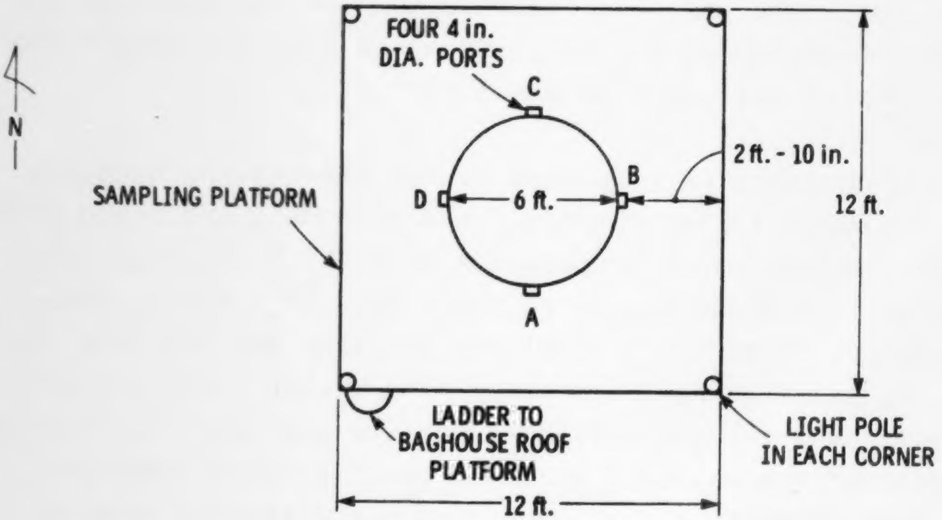
April 15; however, baseline and subsequent waste fuel tests were postponed until April 29 by the Wisconsin DNR after the first day of baseline testing was completed.

## 3.1 LOCATION OF SAMPLING POINTS

The primary sampling site was the exhaust stack of the lime kiln after the baghouse air pollution control device, as shown in Figures 3-1 and 3-2. The stack emerges from the center of the building which contains the eight-module baghouse system. The top of the building is approximately 18.3 m (60 ft) from ground level. The sampling ports are approximately 4.3 m (14 ft) from the top of the baghouse building and 1.1 m (3.5 ft) above the sampling platform. The stack is 1.8 m (6 ft) in diameter with four 10.2-cm (4-in.) sampling ports. The ports are located 0.60 diameters from the nearest upstream disturbance (the juncton of ductwork from the baghouse) and 0.92 diameters from the nearest downstream disturbance (stack outlet). In accordance with EPA Method 1 criteria, a maximum 48-point traverse (24 points in each of two ports, labeled A and B in Figure 3-1) was used for the EPA Method 5 particulate test runs. The port labeled C in Figure 3-1 was used for continuous $NO_x$, $SO_2$, and $O_2$ monitoring; port D was used for continuous CO and THC monitoring and volatile organic sampling train (VOST) sampling.


5 ft. - 6 in.
FOUR 4 in.
DIA. PORTS
2 in.
2 ft. -10 in.
3 ft. - 6 in.
GUARDRAIL
SAMPLING
PLATFORM
LADDER
EXHAUST GAS
FLOW
10 ft.
GUARDRAIL AT
ROOF LEVEL
BAGHOUSE BUILDING
ROOF LEVEL
STAIRS TO
ROOF LEVEL
~60 ft. TO
GROUND LEVEL

(a) SIDE VIEW


N
FOUR 4 in.
DIA. PORTS
C
2 ft. - 10 in.
B
SAMPLING PLATFORM
D
6 ft.
12 ft.
A
LADDER TO
BAGHOUSE ROOF
PLATFORM
LIGHT POLE
IN EACH CORNER
12 ft.

(b) TOP VIEW

Figure 3-1. Exhaust stack sampling locations at Rockwell Lime Company: (a) side view; (b) top view.

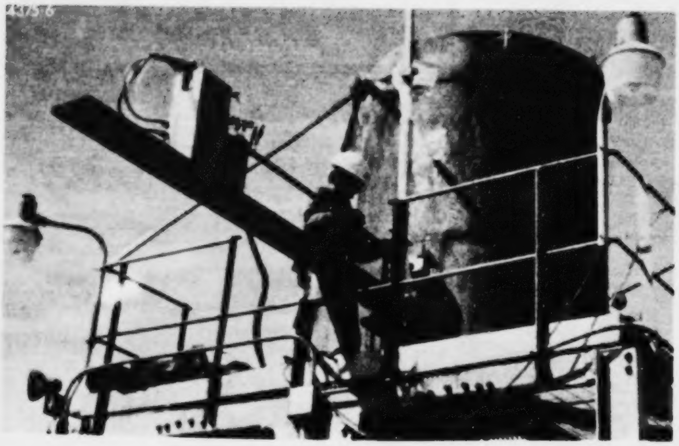


Figure 3-2. Sampling platform and exhaust stack.

Figure 3-1 also illustrates the sampling platform area, which provided ample room. The only modification made at the area was the removal of the upper guardrail at ports A and B to accomodate the EPA Method 5 set-up and train movement.

The laboratory trailers were located at the base of the baghouse building. The main trailer contained EPA Methods 5 and 6 and VOST console boxes, EPA Method 7 preparation area, VOST clean-up and preparation area, and continuous CO, THC, $SO_2$, $NO_x$, and $O_2$ monitoring equipment. A Method 5 umbilical gas line was run from the trailer to the sampling platform along with Teflon lines for EPA Method 6, VOST, and the continuous monitoring systems. The second trailer contained EPA Method 5 clean-up area, process sampling equipment, sample log-in and storage, and miscellaneous reserve sampling equipment.

Other sampling sites used for collecting process samples included the linestone feed conveyor belt to the front end of the kiln (Figure 3-3), the coke feed conveyor belt to the burner, a valve

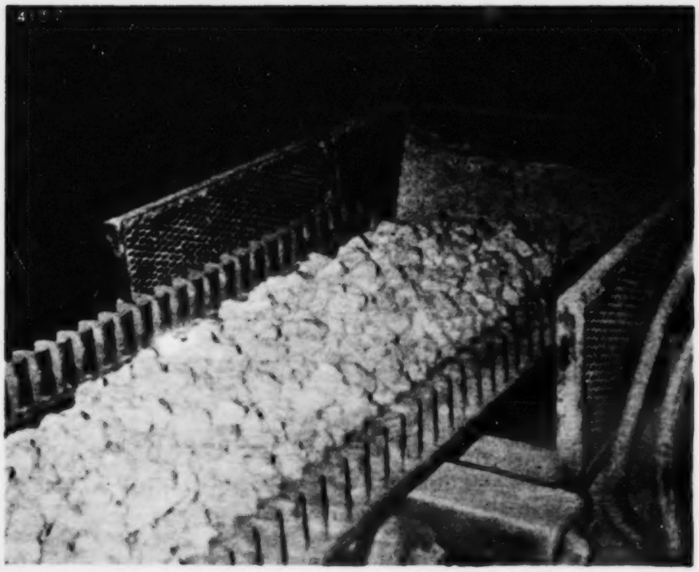


Figure 3-3. Limestone feed conveyor belt.

along the baghouse dust screw conveyor which transported baghouse dust to the storage silo, the lime product as it exited the kiln prior to air cooling, and a valve at a waste fuel pump. Sampling locations are marked with an asterisk in Figure 2-1. Several of the process locations are shown in Figure 3-3, Figure 2-7 (lime product sample point), and Figure 2-8 (waste fuel sample point). The process sites were sampled by compositing four grab samples per day into a daily process sample.

## 3.2 WASTE FUEL ($W_{in}$ FOR POHCs) SAMPLING AND ANALYSIS PROCEDURES

Waste fuel samples were collected from a sampling valve in the waste feed line four times each test day at approximately 2-hour intervals and composited to yield a single 500-mL sample. Samples were contained in a glass bottle with a Teflon-lined cap.

The waste fuel flow rate was determined daily at the plant by two methods. In one method, the difference in storage tankers levels was recorded and the total gallons consumed over various time intervals was calculated. This method became less accurate during runs 7 and 8 when the tanker was at low levels and the change in liquid level depth was high. The second method used a sonic flow meter that was attached to the waste fuel feed pipe. The sonic meter was calibrated against the tanker level difference readings and by pumping into a calibrated bucket over a designated time period. These waste fuel flow rate measurements were supplied by the waste fuel supplier.

Principal organic components were analyzed using a Hewlett-Packard 5985 mass spectrometer, interfaced to a Hewlett-Packard 5840A gas chromatograph (GC/MS) with a 30-m fused silica DB 5 capillary column operated in the split injection mode. The column was programmed at 4°C/min from -10°C to 62°C for the analysis of low-boiling components, and at 8°C/min from 20°C to 300°C for the analysis of high-boiling components. The waste fuel samples were diluted 1:10 in nonane or acetone before introduction into the GC/MS and analyzed in duplicate. Identification was based on the mass spectra (MS) alone or on mass spectra and retention time (MS, RT). Each identified component was quantified relative to a standard of known concentration of that component injected into the GC/MS (major components defined as >1% by weight), or semi-quantified relative to another compound which eluted in the vicinity of that compound (minor components defined as <1%). If a POHC was present at levels less than 1% in the waste, it was quantified against the external standard of the POHC, the same as the major components.

The POHCs chosen for this program and analyzed by GC/MS, split injection mode (SIM), for all waste fuel samples are methylene chloride ($MeCl_2$), methyl ethyl ketone (MEK), 1,1,1-trichloroethane ($CH_3CCl_3$), trichloroethylene (TCE), tetrachloroethylene (Perc), and toluene. Initially, only two POHCs were chosen for the sampling and analyses, TCE and Perc. These two compounds also were spiked into the waste fuel prior to the testing so that detection of the compounds in the stack gas would be less difficult. However, after testing was completed, EPA requested that additional POHCs be added based on the high concentration (i.e., toluene), or the difficulty in destruction (i.e., $MeCl_2$) of particular compounds. A wide-scan analysis to determine the top 20 components in the waste fuel also was conducted by GC/MS for only two fuel samples taken on days 4 and 7.

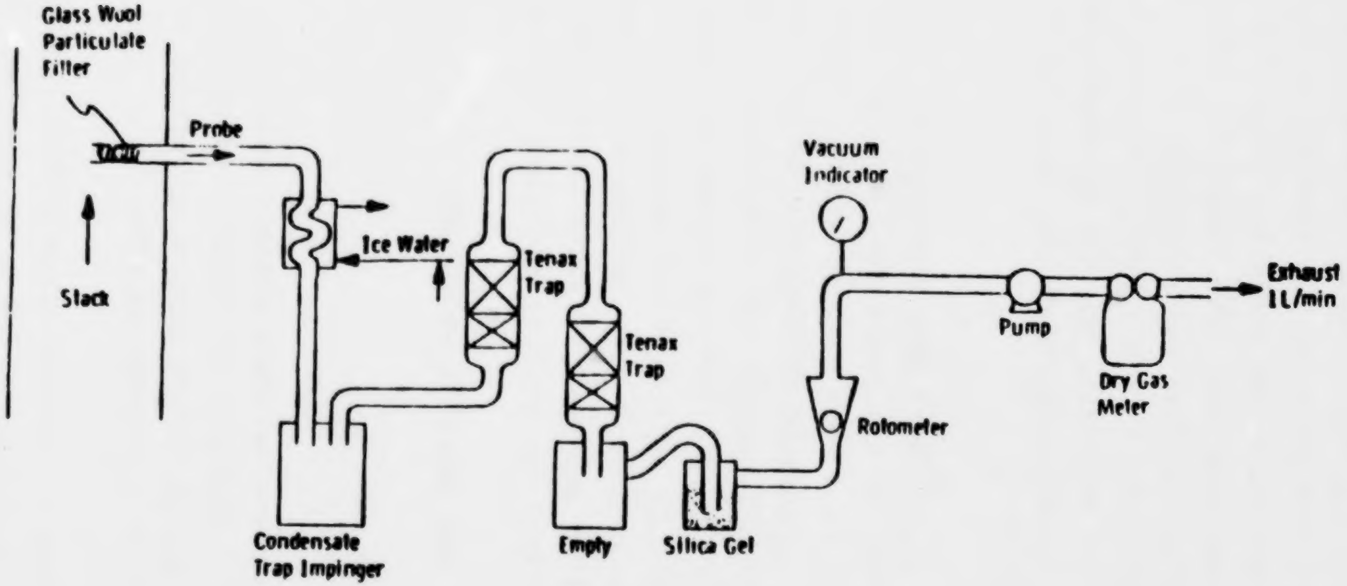
Chlorine and sulfur levels were determined by x-ray fluorescence (XRF). The Btu content of the waste fuel samples was determined by ASTM 240-64, which is a Parr bomb technique. Specific gravity was obtained by the calibrated dilatometer method. Trace metal content of the waste fuel samples was determined by EPA Interim Method 200.7, modified by dilution in xylene followed by ICAP analysis. The ICAP method is an analytical system for simultaneous multielement determinations of trace metals at the sub-ppm levels in solutions. The ICAP analysis of waste fuels used a metal and oil calibration standard for quality assurance (QA) purposes.

## 3.3 STACK SAMPLING AND ANALYSIS PROCEDURES

### 3.3.1 POHCs ($W_{out}$)

POHCs in this program are volatile organic compounds. Collection on an adsorbent resin followed by thermal desorption into a GC/MS system was used to assure enough sample collection to demonstrate destruction and removal efficiencies (DRE).

The EPA Volatile Organic Sampling Train (VOST), shown in Figure 3-4, utilizes a packaged tube of Tenax GC adsorbent resin for primary collection of the volatile organic species.


Glass Wool Particulate Filter
Probe
Stack
Ice Water
Tenax Trap
Tenax Trap
Condensate Trap Impinger
Empty
Silica Gel
Vacuum Indicator
Rotometer
Pump
Dry Gas Meter
Exhaust 1L/min

Note: Tenax traps are 1.6 cm in diameter and 10 cm long.
Tenax traps are mounted vertically.

Figure 3-4. Volatile organic sampling train (VOST).

Another packed tube also containing Tenax GC is placed downstream in the train as a backup to collect any POHC that breaks through from the primary collector. The sampling rate was 1 L/min, and the run time per sample averaged 30 minutes. A new pair of Tenax tubes was used after every 30 L of gas sampled. The tubes were changed after every 30 L of gas sampled for the following reasons:

- At sample volumes greater than 30 liters, some of the very volatile POHCs will break through both the front and backup adsorbent tubes.

- Changing of the tubes allows an initial analysis of one pair of tubes. Analysis of a single pair of tubes lowers the possibility of collecting too much sample and overloading the GC/MS system. However, if the POHCs are not detected or are present at low levels in the single pair, the option exists of combining the contents of the remaining pairs of tubes by thermally desorbing one or more tubes onto one pair of tubes with a concomitant increase in sensitivity.

The required sample volume of 30 L was calculated prior to the test [3] to be within the range of the minimum sample gas volume needed to demonstrate best-case DRE and the maximum sample gas volume to avoid breakthrough of organic compounds.

The Tenax GC (35/60 mesh) was prepared by Soxhlet extraction for 24 hr with methanol/hexane (1:1). The sorbent was then dried in a vacuum oven at 110°C for 6 hr, and packed into 10-cm x 1.6-cm glass tubes. A minimum amount of pre-extracted and oven-dried glass wool was used to hold the sorbent in each of the tubes.

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[3] Day, D. R. Quality Assurance Plan for Hazardous Waste Incineration Study, Rockwell Lime Company. Contract 68-03-3025 SDM-05. U.S. Environmental Protection Agency, Cincinnati, Ohio, April 7, 1983.

The all-Tenax tube contained about 1.6 g of Tenax. The tubes were thermally conditioned prior to use, using a Nutech Model 322 thermal conditioning unit with nitrogen at 200°C for 16 hrs. The conditioning gas (nitrogen) was purified by passing it through a U-trap containing a 5-angstrom molecular sieve with the U-trap immersed in liquid nitrogen. The flow rate of gas through each trap was about 30 mL/min. Following conditioning, each tube was engraved with a unique number, capped with Teflon caps, and transferred to a clean 25-mm x 150-mm screw-cap culture tube ready for sample collection.

The VOST train, shown in Figure 3-4, consisted of:

- A sampling line (1/4-in. Teflon tubing)
- First condenser
- Impinger (for condensate removal)
- Tenax tubes
- Empty impinger
- Silica gel
- Other sampling components (rotameter, pump, dry gas meter)

Before each run, the tubes were connected in the sampling train, and the train was leak checked as in the EPA Method 6 procedure. A leakage rate of ≤2% of the average sampling rate was acceptable. Small sealed charcoal tubes were used during leak checks to prevent contamination of the Tenax tubes by organic vapors in the ambient air. The probe of the train was then placed in the stack, and the train was operated for 30 min at 1 L/min gas sampling rate (gas flow rates in L/min refers to normal conditions of 20°C, 1 atm, dry basis).

During each run, ice water was circulated through the condensers. After about 30 L of gas sample was collected, a post-test leak check was performed. Once again, a leakage rate of ≤2% of the average sampling rate was acceptable. If a leakage rate exceeded 2% the run was not started until the leak was found and sealed and an acceptable leak rate was obtained. Two trains were built on one ring stand so that "back-to-back" runs were made. After the first run was completed, the second train was leak checked before starting the next 30-min sampling period. Upon completion of the second train, a post-leak check was performed, and the two trains were removed from the stack. The two trains were carried to the clean-up area in the sampling trailer, where the used tubes were removed, placed into marked culture tubes, and replaced with new tubes. The clean-up area and tube storage area were located in the sampling trailer and consisted of a 3 ft x 3 ft x 3 ft plastic box under positive pressure to prevent tube contamination.

GC/MS analysis for the six POHCs on the VOST tubes involved the five basic steps listed below and described in detail in the following paragraphs.

- Spiking of the tubes with an internal standard (benzene $d_6$)
- Thermal desorption of the tubes
- Gas chromatography of the desorbed species
- Selected-ion monitoring (SIM) analysis of the POHCs eluting from the chromatographic column (with computer data storage)
- Recall of stored data and calulation of results.

Sample tubes, along with appropriate standards and laboratory blanks, were spiked immediately prior to analysis with 10 ng (10 µg/mL of a 1 µg/mL solution in methanol) of a benzene-$d_6$ internal standard. The tubes were then heated to 175°C for 15 minutes to desorb components onto a 6 ft x 1/4 in. stainless steel 1% SP-100 on 60/80 Carbopack B GC column held at -40°C. The column was programmed to heat at 8°C/min. to 225°C, and then held for 7 minutes. The eluting components were analyzed using a Hewlett-Packard 5983 mass spectrometer operated in the selected ion monitoring mode which scans the major ions of a compound during a specified time frame. Data was collected using a Hewlett-Packard 5937 data system. Four major ions of each analyte and internal standard were monitored. External standards of the compounds of interest were used to determine the correct ion ratios and compound retention times. These standards were prepared at two concentrations by spiking standard solutions of the analytes in methanol onto blank tubes, and then immediately desorbing them in the same manner as the samples.

Concentrations of the analytes were calculated using the internal standard method and the following equation:

$$\text{Amount of analyte} = \frac{\text{AA (samp)}}{\text{AA (std)}} \times \frac{\text{AIS (std)}}{\text{AIS (samp)}} \times \text{AMT (std)}$$

where AA (samp) = area of analyte in the sample
AA (std) = area of analyte in the external standard
AIS (std) = area of internal standard in the standard run
AIS (samp) = area of internal standard in the sample run
AMT (std) = amount of analyte in external standard (wt)

The mass of a specific compound was then used along with sample meter conditions (i.e., sample volume and temperature) and stack conditions (i.e., stack flow rate) to calculate the mass emission rate for a given compound.

### 3.3.2 Stack Gas Velocity, Flow Rate, Dry Molecular Weight and Moisture Content

The procedure and equipment used to determine stack gas conditions followed the specifications in EPA Methods 1 through 4 outlined in the Federal Register (40 CFR part 60). The location of the traverse points (Method 1) was determined during setup days before testing. Because upstream and downstream disturbances were close to the sample ports (within one stack diameter), the maximum 48 points were required for Method 5 sampling. In addition, a preliminary velocity traverse (Method 2) was performed to obtain a preliminary stack velocity and determine if conditions were acceptable for sampling. Method 2 also was performed each day. Methods 3 and 4 were performed only during Method 5 runs. $CO_2$ and $O_2$ concentrations and dry molecular weight calculations (Method 3) were determined using Fyrite analyzers. $O_2$ stack concentration also was monitored continuously and can be compared to Fyrite results as a QA/QC check. Method 5 back-half impingers were used for stack moisture determination (Method 4).

### 3.3.3 Particulate Matter

The procedure and equipment used for quantitative particulate collection met the specifications outlined in EPA Method 5. The flue gas exhaust stack was traversed for 48 points (12 point each on two perpendicular diameters) at 2 min./point for representative sample collection. Mass emissions were collected over a period of about 1.5 hours from at least 39 dry standard cubic feet (dscf) of exhaust gas. As a comparison, a sample gas volume of >30 dscf is required under the New Source Performance Standards (NSPS) for cement kilns.

A schematic diagram of the EPA Method 5 sampling train is shown in Figure 3-5. The front half of the train (filter and probe rinse) was used to determine particulate loading. The back half of the train (impinger section) was used for moisture determination and HCl sampling (see HCl procedure discussion).

The Method 5 train probe consisted of a 6-ft glass-lined probe. Reeve Angel Type 934 AH filters were used. A handheld computer program was used to set up the nomograph for calculating the K factor during Method 5 testing.

After sampling, the filters were desiccated and then weighed using a calibrated analytical balance. The filters were dessicated and weighed again until constant weight was achieved (weight was within

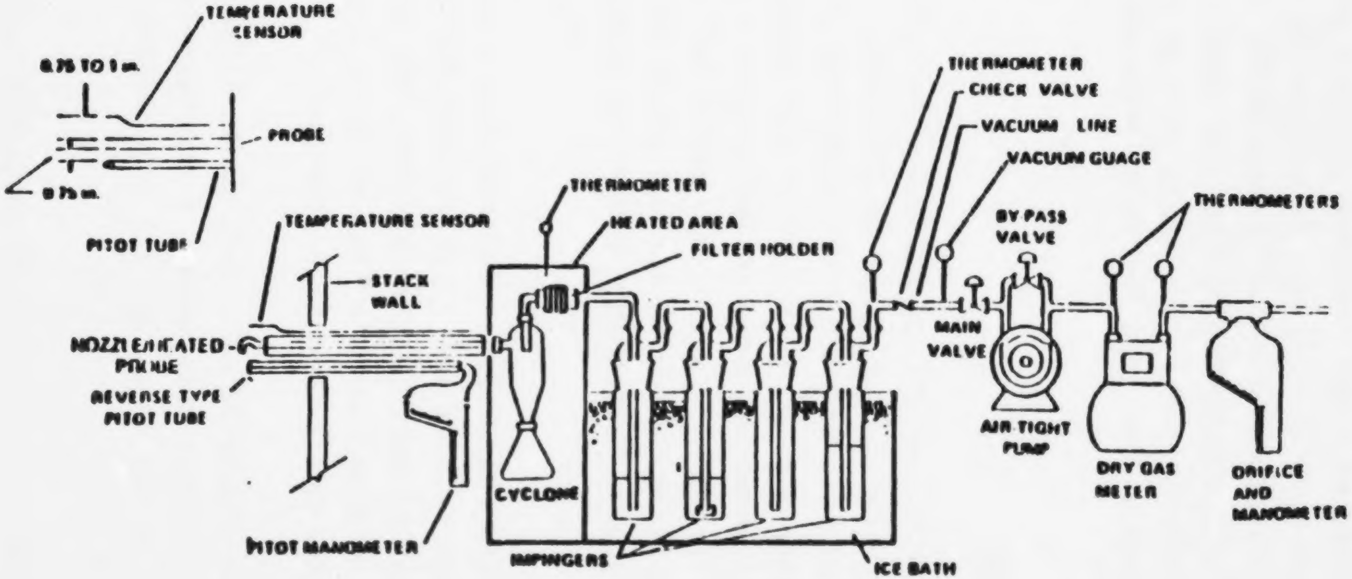
TEMPERATURE SENSOR
0.75 TO 1 in.
PROBE
0.75 in.
PITOT TUBE
TEMPERATURE SENSOR
STACK WALL
NOZZLE HEATED PROBE
REVERSE TYPE PITOT TUBE
PITOT MANOMETER
THERMOMETER
HEATED AREA
FILTER HOLDER
CYCLONE
IMPINGERS
ICE BATH
THERMOMETER
CHECK VALVE
VACUUM LINE
VACUUM GUAGE
BY PASS VALVE
MAIN VALVE
AIR TIGHT PUMP
THERMOMETERS
DRY GAS METER
ORIFICE AND MANOMETER

Figure 3-5. Particulate sampling train for EPA Method 5.

1% of previous weighing). The weight of collected particulate matter was determined by subtracting the initial weight (before sampling) from the final weight (after sampling) of the filter. Several filters were used as QC blanks. Also, acetone probe rinse blanks were taken as QC blanks. An HP-85 computer was taken to the field, and all EPA Method 5 data were stored and isokinetic calculations were made from the Method 5 program at the end of each day. The concentration of particulate matter in the stack gas for the samples collected was computer-calculated from the recorded sampling and analytical data using the HP-85 upon return to the Dayton Laboratory.

### 3.3.4 Metals on Particulate Matter

Particulate matter samples collected by Method 5 (evaporated probe rinse and filter) were analyzed for trace metals using inductively coupled argon plasma emission spectroscopy (ICAP) following EPA interim Method 200.7. The ICAP method is an analytical system for simultaneous multielement determinations of trace metals at the sub-ppm level in solutions. The basis of the method is atomic emission promoted by coupling the sample, nebulized to form an aerosol using high temperature argon plasma produced by the passage or argon through a radio-frequency field. Twelve EPA Method 5 runs were made, of which nine runs were submitted for metals. The remaining three runs were analyzed for dioxins on particulates as described in the next subsection. Blank filters were analyzed by ICAP to determine if any contamination existed due to the filter.

### 3.3.5 Dioxins on Particulate Matter

EPA Method 5 particulate matter samples (filters only) collected from three runs (one baseline and two waste fuel condition samples) were analyzed for dioxin isomers. The analytical procedure followed, in general, the extraction and cleanup described in the US EPA Region 7 Protocol for the Determination of TCDD in Soil and Sediment. However, the acid pretreatment and toluene Soxhlet extraction described by Hutzinger was used to remove the chlorinated dioxins from the matrix. The samples were cleaned up with Option A followed by basic alumina column chromatography as described in the Region 7 method. The analyses were performed on a general purpose 30m x 0.25mm DB-5 fused silica column from J&W Scientific.

The quantitation was based upon an internal standard method. Response factors were calculated for representatives of tetra through octachlorodibenzo-p-dioxin (OCDD) compared to 13C(12)-2378 TCDD. These response factors were used to calculate detection limits in the manner described in the Region 7 method. Analyses for tetra through octachlorinated dioxins were conducted on a low resolution quadrupole mass spectrometer (Finnigan 1020). The detection limits in the Method 5 filter samples were all well below 1 ng per filter except for OCDD. The detection limits and data submitted are the best of three attempts.

### 3.3.6 Hydrogen Chloride

HCl was collected in the back half of the EPA Method 5 sampling train in an absorbing collection medium of 0.5 M sodium acetate (NaoAc). The hydrogen chloride was trapped in the dilute solution, forming ionic sodium chloride. The sample was analyzed using a chloride ion specific electrode (SIE) and an expanded scale millivolt/pH meter. This sampling and analysis procedure is a modification of the NIOSH S246 procedure for HCl in air, scaled up for use on midget impinger sizes.

The impinger train, which was immersed in an ice bath during sampling, consisted of one bubbler and four impingers connected by glass U-tubes and clamps. Bubbler No. 1 and impinger No. 2 each contained 100 mL of sodium acetate absorbing solution. Impinger No. 3 contained 150 mL of 3% $H_2O_2$ solution, which removed $SO_2$ from the sample stream and protected the meter and pump. Impinger No. 4 was a dry impinger that functioned to remove moisture and/or catch carryover. Impinger No. 5 contained 200 to 300 g of 6-mesh to 16-mesh silica gel.

For analysis, the collected sample was adjusted to a pH of 5 with acetic acid, and the chloride SIE reading was taken and compared to an analytical standards curve prepared from a series of five chloride standard solutions. Results were reported as μg Cl per sample, which were used to calculate stack gas concentration.

### 3.3.7 Continuous Monitoring - CO and THC

Continuous monitoring for carbon monoxide and total hydrocarbons was conducted during all sampling days. The continuous monitoring systems are described as follows:

| Pollutant | Manufacturer | Model No. | Analytical technique |
|---|---|---|---|
| CO | Beckman | 865 | Nondispersive infrared spectrometer (EPA Method 10) |
| THC | Beckman | 402 | Flame ionization detector |

#### 3.3.7.1 Beckman Model for CO--

The Beckman analyzer measures CO concentrations continuously in the stack gas. A light is passed through a cell containing the stack gas sample. When the infrared radiation strikes a CO molecule, the light energy is absorbed and will be excited and produce energy. Light also is passed through a reference cell, which produces energy. A detector measures the difference between the sample and reference energy produced, a pressure difference develops, and a diaphragm moves to plot the CO concentration on the chart recorder. The greater the amount of CO in the gas, the greater the pressure difference.

Interferences caused by water and carbon dioxide were removed using silica gel and ascarite, respectively. Calibration at zero, 30%, 60%, and 90% of the 0 to 1,000-ppm range were performed before, several times during, and after each day.

### 3.3.7.2 Beckman Model for THC--

Total hydrocarbons were measured using a continuous flame ionization detector (FID). The FID system burns the sampled stack gas, which emits ions and electrons formed in the flame. The ions and electrons enter the electrode gap and decrease the gap resistance, thus permitting a current to flow in the external circuit, which is converted to a concentration.

Calibration gases at zero, 5%, 19%, and 50% of the 0 to 100-ppm scale were used to calibrate the FID daily before, several times during, and after each run.

### 3.3.7.3 Sample Handling System--

The system used to obtain samples for CO and THC continuous monitoring is shown in Figure 3-6 and consists of:

- In-line sample probe with filter
- Heated sample lines
- Heated manifold
- Aspirator
- Air compressor
- Silica gel container
- Ascarite container

Sample was collected through a stainless steel probe with a particulate filter. An air compressor/aspirator system was used to sample the stack gas for the CO and THC analyzers.

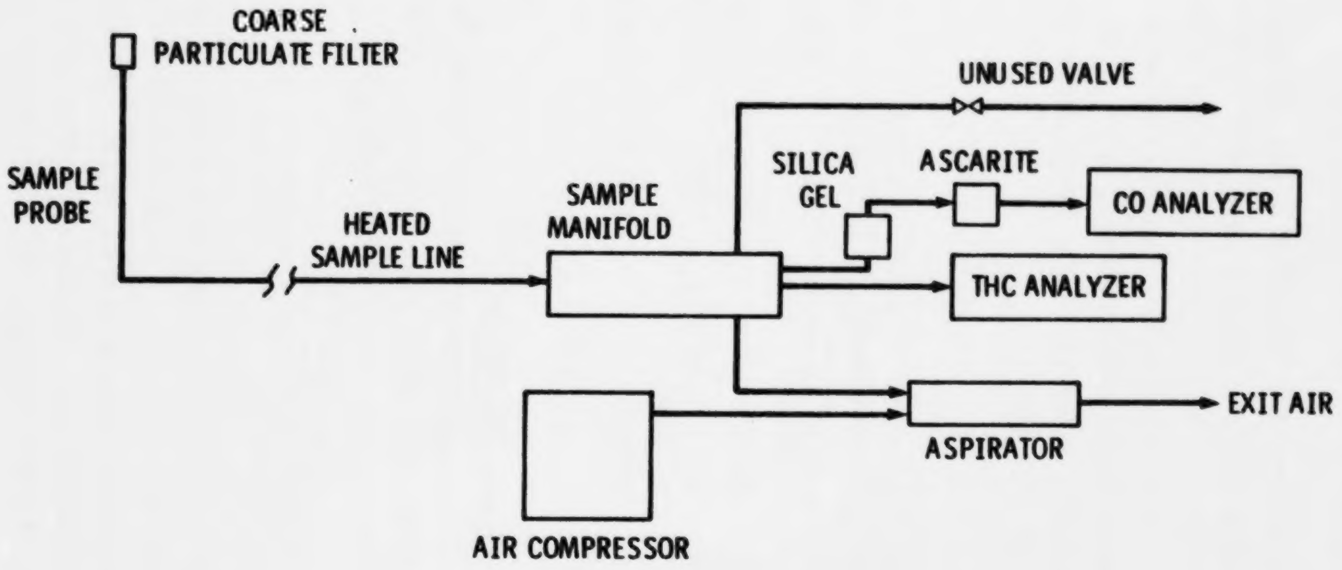
COARSE
PARTICULATE FILTER
SAMPLE
PROBE
HEATED
SAMPLE LINE
SAMPLE
MANIFOLD
UNUSED VALVE
SILICA
GEL
ASCARITE
CO ANALYZER
THC ANALYZER
AIR COMPRESSOR
ASPIRATOR
EXIT AIR

Figure 3-6. Sample handling system for continuous monitoring of CO and THC.

### 3.3.8 Continuous Monitoring - Sulfur Dioxide, Nitrogen Oxides, and Oxygen

Continuous monitoring for the stack gas emission of sulfur dioxide, nitrogen oxides, and oxygen was conducted during Days 2 through 8. On Day 1, only nitrogen oxides continuous monitoring was performed. The continuous monitoring work was subcontracted by MRC to Entropy Environmentalists, Inc. (Days 2 through 8) and PEDCo Environmental (Day 1). The continuous monitoring systems are described as follows:

| Pollutant | Day used | Manufacturer | Model No. | Analytical technique |
|---|---|---|---|---|
| $NO_x$ | 1 | Thermo Electron (TECO) | 10A | Chemiluminescence |
| $NO_x$ | 2-8 | Beckman | 951A | Chemiluminescence |
| $SO_2$ | 2-8 | Thermo Electron (TECO) | 40 | Pulsed fluorescence |
| $O_2$ | 2-8 | Teledyne | 320P-4 | Electrocatalytic fuel cell |

#### 3.3.8.1 TECO Model for $NO_x$--

The TECO 10A analyzer measured NO concentrations by mixing the gas sample with ozone ($O_3$) in a flow reactor. The NO and $O_3$ form $NO_2$ and $O_2$ according to the following reaction:

$$NO + O_3 \rightarrow NO + O_2 + \lambda V$$

Light emission results when the electronically excited molecules revert to their ground state. A photomultiplier tube responds to this light and generates a signal which is linearly proportional to the NO concentration. To measure $NO_x$, the gas sample is passed through an $NO_2$-to-NO thermal converter prior to analysis of the NO. $NO_2$ is determined by difference. The gas sample was obtained from the MRC gas manifold (described in Section 3.3.7) and passed through a stainless steel condenser and glass wool filter before entering the analyzer. The sample flow rate and pressure were maintained at 0.05 std $m^3$/hr (1.75 scfh) and 25.4 cm (10 in.) Hg vacuum, respectively, during calibration and sampling. The instrument field stabilized at least 24 hours prior to data collection. A three-point calibration was carried out before and after the day's sampling with additional span and zero checks during the sampling period.

### 3.3.8.2 Beckman Model for $NO_x$--

The Beckman Model 951A NO/$NO_x$ analyzer used on Days 2 through 8 continuously determined the concentration of nitric oxide (NO) or oxides of nitrogen ($NO_x$) in the stack. In the determination of NO, the sample is quantitatively converted to $NO_2$ by gas phase oxidation with molecular ozone produced within the analyzer. In this reaction, the $NO_2$ molecules are elevated to an electronically excited state, and then immediately reverted to a non-excited ground state. This reversion is accompanied by emission of photons, which impinge on a photomultiplier detector, and generate a low level DC current. The current is then amplified and used to drive a front panel meter and a data recorder.

For the determination of $NO_x$, the analyzer utilizes the same principles as it does for determination of NO, except that before the sample enters the reaction chamber, it is routed through a converter, where the $NO_2$ is dissociated to form NO. The $NO_x$ concentration seen by the instrument includes the contributions of both the NO in the effluent and the NO resulting from the dissociation of $NO_2$.

The Beckman 951A NO/$NO_x$ is equipped with seven ranges: 10, 25, 100, 250, 1,000, 2,500, and 10,000 ppm of NO or $NO_x$, full scale.

The $NO_x$ analyzer was calibrated for its 0 to 1,000 ppm range. The zero was first adjusted using air as zero gas. A high level span gas was then injected into the monitor, and the span was adjusted to obtain a monitor reading which agreed with the calibration gas value. After the zero and span were adjusted, low and mid level gases were injected to verify the monitor's linearity. All upscale calibration gases were mixtures of NO in $N_2$.

### 3.3.8.3 TECO Model for $SO_2$--

The TECO Series 40 $SO_2$ analyzer continuously determined the concentration of $SO_2$ in the stack. Because this analyzer is sensitive to water vapor interference, an internal permeation tube type dryer is employed to remove any residual water vapor from the sample stream.

Within the analyzer, the gas sample is submitted to a monochromatic source of pulsed ultraviolet illumination. This high intensity UV source energizes the $SO_2$ molecules, causing them to emit an $SO_2$-specific illumination. The emitted light, which is approximately proportional to the concentration of $SO_2$ present in the sample, passes through a photomultiplier tube. The output of the photomultiplier tube is electronically amplified to provide a front panel meter reading and an output for a data recorder.

To compensate for the small non-linearity of the analyzer output, a calibration curve is generated from a zero gas and two or three upscale calibration gas mixtures of $SO_2$ in air. All concentration data are read from this calibration curve.

The background gas in most typical combustion sources has a quenching effect on the fluorescence within the analyzer. The two gases which contribute most to this quenching effect are $CO_2$ and $O_2$. To compensate, concentration data must be divided by the appropriate quenching factor.

Using air as zero gas for $SO_2$, the zero pot was adjusted to give an analyzer output of zero. The high level span was then injected into the monitor, and the monitor span was set. The analyzer was calibrated for its 0 to 1,000 ppm $SO_2$ range. Since concentrations slightly in excess of 1,000 ppm were expected, the monitor's gain was adjusted to give a 840 ppm response to a 923 ppm span gas, thus making the monitor's effective range 0 to 1,100 ppm. After the zero and span were adjusted, the monitor's responses to mid- and low-level gases were documented and used to construct a calibration curve. This curve was used to correct for the analyzer's slight non-linearity. All upscale calibration gases were mixtures of $SO_2$ in air.

### 3.3.8.4 Teledyne Model for $O_2$--

The Teledyne Model 320P-4 utilizes Teledyne's patented micro-fuel cell, which consumes $O_2$ from the atmosphere surrounding the measuring probe. The comsumption of $O_2$ generates a linearly proportional electrical current. This current is then amplified and used to drive a built-in front panel meter with scales of 0 to 10% and 0 to 25%.

The $O_2$ analyzer was calibrated for both the 0 to 10% $O_2$ and 0 to 25% $O_2$ ranges. The Teledyne analyzer has no zero adjustment. Air was used as the high level span gas (20.9% $O_2$). After the span adjustment was made, the intermediate value gas was injected, and the monitor response was recorded on the calibration data sheet. All $O_2$ calibration gases were mixtures of $O_2$ in $N_2$. A $NO_x$ calibration gas ($NO/NO_2$) was used to check the $O_2$ analyzer's zero response.

### 3.3.8.5 Sample Handling System--

The sample handling system provides a continuously conditioned sample gas flow for each of the gas analyzers. Two different sample acquisition systems were used to supply the continuous monitors during Days 2 through 8. On Days 2 and 3, the sample was taken from MRC's sampling manifold (described in Section 3.3.7). The sample flow requirements for the $SO_2$, $NO_x$, and $O_2$

systems were substantially greater than for the CO and THC systems, causing excessive pressure drop through the sample line. This situation resulted in marginal sample flow rates for all monitors. Therefore a separate sample acquisition system was set up and used to supply sample gas for $SO_2$, $NO_x$, and $O_2$ monitoring for Days 4 through 8. The major sample handling components are as follows:

- In-stack probe
- In-stack course particulate filter
- Heated sample line
- Ice bath condenser
- Gas transport tubing
- Sampling pump
- Flow distribution system
- Fine particulate filters

Figure 3-7 illustrates the sample handling system. The probe was position in the stack such that the inlet was about one inch from the inlet to MRC's continuous monitoring probe.

The in-stack probe was constructed of 316 stainless steel and was heated by a Nichrome wire wrapped between its inner and outer liners. A variac was used to control the electrical power supplied to the heater. The probe was equipped with an enlarged tip packed with borosilicate glass wool which served as an in-stack particulate filter.

a 10-ft length of 3/8-in. OD Teflon tubing, wrapped with Nichrome wire and insulation, served as the heated sample line which connected the probe to the ice bath condenser. Electrical power to the sample line heater was controlled by a variac.

The condenser consisted of a coil of 3/8-in. OD Teflon tubing followed by glass condensate traps, all immersed in an ice bath

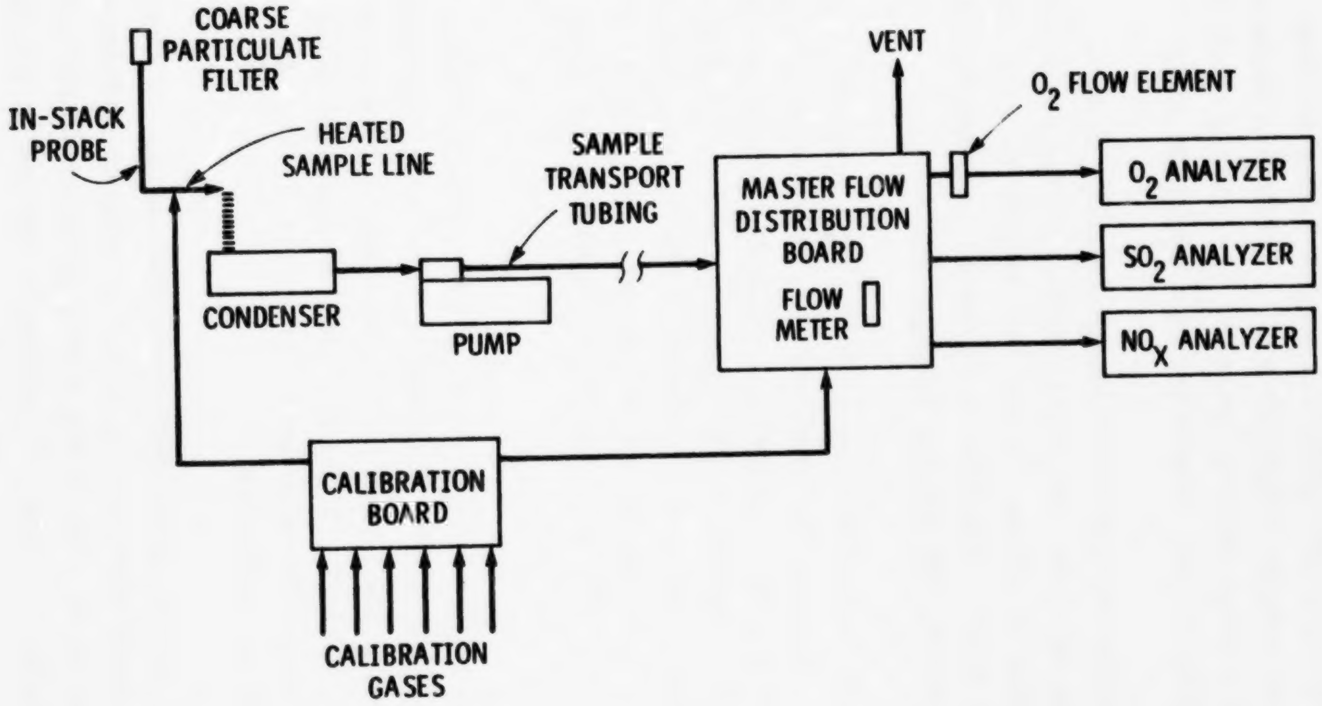
COARSE PARTICULATE FILTER
IN-STACK PROBE
HEATED SAMPLE LINE
CONDENSER
PUMP
SAMPLE TRANSPORT TUBING
VENT
MASTER FLOW DISTRIBUTION BOARD
FLOW METER
$O_2$ FLOW ELEMENT
$O_2$ ANALYZER
$SO_2$ ANALYZER
$NO_X$ ANALYZER
CALIBRATION BOARD
CALIBRATION GASES

Figure 3-7. Sample handling system for $SO_2$, $NO_x$, and $O_2$ continuous monitoring.

to minimize contact between the sample and the condensate. The sample exiting the condenser had a moisture content of about 0.6% and was transported through unheated smaple lines without further condensation. The sample transport line, which was made of unheated 3/8-in. OD Teflon tubing, carried the sample from the condenser to the Teflon-lined, diaphram-type sampling pump.

The flow distribution system received gas either from the sample acquisition system or from the calibration gas manifold board. The flow system then distributed the gas sample to each individual analyzer and to a vent. The vent allowed an increased sampling flow rate, which minimized system response time.

Fine particulate filters were installed at the monitors' sample inlet in order to protect the analyzers in the event of particle entrainment at the analyzer location. The $NO_x$ analyzer was equipped with with a 316 stainless steel filter utilizing a 7-micron sintered stainless steel element. The $O_2$ analyzer was equipped with a factory installed polyester wool filter. The $SO_2$ analyzer's filter had been removed for service and was not employed during this testing.

Leak checks were performed each morning by closing off the three-way valve at the inlet to the condenser. The sampling pump was then used to evacuate the lines until the sample flow meter ball came to a complete rest on its seat. Any in-leakage would have been indicated by a slight hopping of the ball on its seat. The system was found to be leak-free.

In addition to the daily leak check described above, a system calibration check was performed on Day 7. The results indicated that the system was leak-free. This check was performed one time only and used one gas only, in order to conserve the calibration gases.

After the initial calibration and leak check procedures wee completed, monitoring system was operated continuously except for interruptions for monitor calibration checks. The $SO_2$ analyzer was kept on the 0 to 1,000 ppm range (documented on the charts as 0 to 1,100 ppm range), except during process upsets, when $SO_2$ concentrations soared. During these excursions, in an effort to track the $SO_2$ concentrations, the monitor was switched to the 0 5,000 ppm range. At the time of this testing, there was no $SO_2$ calibration gas available with a high enough $SO_2$ concentration to effectively calibrate this range. Consequently, there is an inherent uncertainty associated with $SO_2$ data collected during these excursions, which typically lasted from one to three minutes.

The $O_2$ analyzer was operated on either the 0 to 10% or the 0 to 25% scale, depending on where the $O_2$ trace least interfered with the other two traces on the strip chart.

The $NO_x$ analyzer was operated on the 0 to 1,000 ppm range throughout the testing program.

### 3.3.9 Sulfur Dioxide

Six sulfur dioxide samples were collected and analyzed using EPA Method 6. Three 20-minute samples were collected on Days 1 and 2 as the only $SO_2$ stack measurement made. Three additional runs were made on Days 3 and 7 to compare with the continuous systems. All Method 6 sampling used one midget bubbler, containing 15 mL of 80% isopropanol (IPA), and four midget impingers, two containing 15 mL of 30% $H_2O_2$, the third empty, and the fourth containing silica gel to protect the metering equipment. Each Method 6 sample consisted of at least 0.5 standard cubic feet of stack gas. Analytical results were used to calculate lb/SCF stack gas concentration following Method 6 procedures, and stack volumetric data from Methods 1 through 5 were used to calculate $SO_2$ mass emission rates (lb/hr).

### 3.3.10 Nitrogen Oxides

One EPA Method 7 run was used to determine emissions of nitrogen oxides to compare with the continuous monitoring system. Four grab samples were collected over an approximately 1-hour period on Day 3 and analyzed individually. Sampling procedures included pipetting 25 mL of absorbing reagent (dilute $H_2O_2/H_2SO_4$) into ball flasks, evacuating the flasks to vacuum, drawing a stack gas sample into the flask, adding 1M NaOH to a pH of approximately 2, and recording ambient conditions, time of day, and flask pressures. Analyses were completed using the spectrophotometric acid procedure and were reported in μg $NO_2$ per sample. Analytical results were used to calculate concentration in lb/scf and stack conditions used to calculate mass emissions in lb/hr of $NO_x$.

### 3.3.11 Process Samples

Petroleum coke, limestone feed, lime product, baghouse dust, and waste fuel process samples were colleced four times per day at approximately 2-hour intervals and composited to make one daily sample. Chlorine was analyzed in all samples, and sulfur was analyzed in all samples except limestone feed using x-ray fluorescence (XRF) based on externally prepared standards. All process samples also were analyzed for trace metals by ICAP, using EPA Interim Method 200.7, as previously described. Coke samples also were analyzed for Btu and ash content using ASTM standard methods. Two additional baghouse dust samples were taken simultaneously with Method 5 runs on Days 3 and 5 and analyzed for dioxins to correlate with the dioxins on Method 5 particulate filter samples (see Section 3.3.5). Waste fuel samples were analyzed for POHCs, wide-scan organics, metals, chlorine, sulfur, Btu, and specific gravity as previously described in Section 3.2.

# SECTION 4

# DEMONSTRATION TEST BURN RESULTS

## 4.1 TEST BURN RUNS

Sampling was conducted on eight days, each day representing a run. Thus, Run No. 1 was conducted on the first day of testing, Run No. 2 on the second day and so on as shown below:

| No. | Date, 1983 | Condition |
|---|---|---|
| 1 | Friday, 4/15 | Baseline |
| 2 | Friday, 4/29 | Baseline |
| 3 | Saturday, 4/30 | Baseline |
| 4 | Monday, 5/2 | Waste fuel |
| 5 | Tuesday, 5/3 | Waste fuel |
| 6 | Wednesday, 5/4 | Waste fuel |
| 7 | Thursday, 5/5 | Waste fuel |
| 8 | Friday, 5/6 | Waste fuel |

For Run Nos. 4 and 7, the waste fuel rate was changed at midday such that two waste fuel conditions were performed on each of those days.

Samples taken during each run are labeled by the run number. Multiple samples taken during each run are designated by letters following the run number (e.g., Run No. 2B represents the second day, second sample taken).

Waste fuel was added only during daylight hours, generally between 7:00 a.m. and 5:00 p.m. During evening hours, the kiln burned natural gas and coke as fuel. After switching from natural gas to waste fuel each morning, the kiln was allowed to stabilize prior to sampling. Typically, the kiln operated normally; however, on several occasions, kiln temperatures and product rates increased above normal conditions. These excurtions and their causes and effects on emissions are discussed in Section 5.4.

## 4.2 STACK SAMPLING RESULTS

### 4.2.1 Stack Conditions

Stack conditions, as measured by EPA Methods 1 through 4 during Method 5 sampling, are shown in Table 4-1. Stack velocities ranged from 4.9 to 6.0 m/s (16.0 to 19.7 ft/s) and averaged 5.4 m/s (17.6 ft/s), at an average temperature of 202°C (395°F) and an average moisture content of 6.77%. Actual stack flow rates ranged from 791 to 975 $m^3$/min (27,950 to 34,450 $ft^3$/min) and averaged 870 $m^3$/min (30,720 $ft^3$/min). Although Run No. 5B is invalid, on the basis of the definitions is the Federal Register for the case of a poor leak check caused by a crack in the glass probe, data for that run will be presented in this report.

### 4.2.2 Particulate and HCl Emissions

Particulate emissions remained at low levels during all runs. As shown in Table 4-2, average particulate emissions were 0.89 kg/hr (1.97 lb/hr) for baseline conditions and 1.01 kg/hr (2.23 lb/hr) for waste fuel conditions.

The average particulate stack gas concentration was 29.8 mg/dncm ± 4.7 mg/dncm (95% confidence limits) (0.013 grains/dscf ± 0.002 grains/dscf) for baseline conditions and 36.6 mg/dncm ± 8.0 mg/dncm (95% confidence limits) (0.016 grains/dscf ± 0.004 grains/

# TABLE 4-1. STACK CONDITIONS

| Run No. | Stack temperature °C | Stack temperature °F | Stack static pressure cm $H_2O$ | Stack static pressure in. $H_2O$ | Water content, % $H_2O$ | Percent[a] $CO_2$ | Percent[a] $O_2$ | Dry molecular weight, kg/kg-mole (lb/lb mole) | Stack velocity m/s | Stack velocity ft/s | Stack flow rate (dry) $\frac{dscm}{min}$ | Stack flow rate (dry) $\frac{dscf}{min}$ | Stack flow rate (actual) $\frac{m^3}{min}$ | Stack flow rate (actual) $\frac{ft^3}{min}$ |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1A | 210 | 410 | 0.051 | 0.02 | 5.94 | 25 | 8.6 | 32.34 | 6.0 | 19.7 | 543 | 19,170 | 975 | 34,450 |
| 1B | 211 | 416 | 0.051 | 0.02 | 7.86 | 25 | 5.5 | 32.20 | 5.8 | 19.1 | 514 | 18,150 | 944 | 33,340 |
| 2 | 201 | 393 | 0.051 | 0.02 | 6.41 | 24.0 | 8.8 | 32.19 | 5.8 | 19.0 | 534 | 18,880 | 943 | 33,320 |
| 3 | 199 | 389 | 0.051 | 0.02 | 6.15 | 20.2 | 8.3 | 31.56 | 5.0 | 16.4 | 458 | 16,190 | 805 | 28,430 |
| 4 | 195 | 383 | 0.051 | 0.02 | 6.87 | 21.7 | 6.5 | 31.73 | 5.2 | 17.1 | 472 | 16,680 | 846 | 29,900 |
| 5A | 196 | 384 | 0.051 | 0.02 | 5.91 | 21.2 | 5.8 | 31.62 | 4.9 | 16.0 | 451 | 15,940 | 791 | 27,950 |
| 5B[b] | 198 | 388 | 0.051 | 0.02 | 6.82 | 22.2 | 5.2 | 31.75 | 4.9 | 16.1 | 449 | 15,860 | 798 | 28,210 |
| 6A | 203 | 397 | 0.051 | 0.02 | 7.13 | 21.3 | 6.0 | 31.65 | 5.1 | 16.6 | 455 | 16,080 | 824 | 29,130 |
| 6B | 205 | 400 | 0.051 | 0.02 | 7.50 | 22.7 | 4.3 | 31.80 | 5.5 | 18.0 | 488 | 17,230 | 890 | 31,460 |
| 7A | 206 | 403 | 0.051 | 0.02 | 6.48 | 22.0 | 3.8 | 31.67 | 5.8 | 19.0 | 526 | 18,590 | 938 | 33,160 |
| 7B | 197 | 387 | 0.051 | 0.02 | 7.28 | 22.3 | 1.3 | 31.63 | 5.0 | 16.2 | 456 | 16,070 | 805 | 28,430 |
| 8 | 201 | 393 | 0.051 | 0.02 | 6.91 | 22.3 | 0.7 | 31.60 | 5.4 | 17.8 | 494 | 17,460 | 880 | 31,100 |

[a] By Fryrite Method.

[b] Post leak check out of specs due to crack in glass probe.

## TABLE 4-2. PARTICULATE AND HCl STACK GAS CONCENTRATIONS

| Run No. | Run condition | Sample volume | | Particulate concentration | | Particulate emissions rate | | HCl concentration, ppm | HCl emissions rate | | Percent isokinetic |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | dncm | dscf | mg/dncm | grains/dscf | kg/hr | lb/hr | | kg/hr | lb/hr | |
| 1A[a] | Baseline | 1.58 | 55.7 | 35.0 | 0.015 | 1.14 | 2.52 | 0.74 | 0.037 | 0.084 | 115.6 |
| 1B | Baseline | 1.42 | 50.2 | 24.7 | 0.011 | 0.76 | 1.69 | 1.42 | 0.071 | 0.156 | 109.9 |
| 2 | Baseline | 1.38 | 48.6 | 31.1 | 0.014 | 1.00 | 2.21 | 1.83 | 0.091 | 0.200 | 102.3 |
| 3 | Baseline | 1.19 | 41.9 | 24.0 | 0.010 | 0.66 | 1.45 | 3.88 | 0.169 | 0.371 | 103.1 |
| 4 | Waste fuel | 1.20 | 42.3 | 26.4 | 0.012 | 0.75 | 1.66 | 2.54 | 0.115 | 0.253 | 100.8 |
| 5A | Waste fuel | 1.14 | 40.1 | 24.9 | 0.011 | 0.68 | 1.49 | 4.04 | 0.172 | 0.380 | 100.1 |
| 5B[b] | Waste fuel | 1.12 | 39.7 | 28.7 | 0.013 | 0.78 | 1.71 | 4.96 | 0.213 | 0.469 | 99.6 |
| 6A | Waste fuel | 1.17 | 41.2 | 35.8 | 0.016 | 0.98 | 2.16 | 4.79 | 0.209 | 0.460 | 102.0 |
| 6B | Waste fuel | 1.23 | 43.4 | 35.0 | 0.015 | 1.03 | 2.26 | 5.39 | 0.253 | 0.557 | 100.2 |
| 7A | Waste fuel | 1.29 | 45.4 | 35.6 | 0.016 | 1.13 | 2.49 | 2.98 | 0.149 | 0.329 | 97.2 |
| 7B | Waste fuel | 1.15 | 40.8 | 48.7 | 0.021 | 1.34 | 2.94 | 5.95 | 0.261 | 0.575 | 100.8 |
| 8 | Waste fuel | 1.27 | 44.7 | 47.5 | 0.021 | 1.41 | 3.12 | 4.73 | 0.220 | 0.493 | 101.8 |

[a] Run invalid as percent isokinetic was greater than 100% ± 10%.

[b] Bad leak check; run invalid.

dscf) for waste fuel conditions. These concentrations are typical of lime kiln particulate emissions based on values listed in the McIlvaine Manual that range from 0.007 to 0.04 grains/dscf [4].

HCl emissions averaged 0.092 kg/hr ± 0.0103 kg/hr (95% confidence limits) (0.203 lb/hr ± 0.226 lb/hr) for baseline conditions and 0.199 kg/hr ± 0.061 kg/hr (0.438 lb/hr ± 0.133 lb/hr) for waste fuel conditions as shown in Table 4-2. These results show an increase in HCl emissions from baseline to waste fuel conditions, as expected, due to the additional chlorine input to the kiln from the waste fuel.

The percent isokinetics ranged from 97.2% to 115.6%. Run No. 1A was invalid because it was not within the 100% ± 10% isokinetic criteria specified in the EPA Method 5 sampling procedures. However, data for Run No. 1A is presented as additional information. All other runs were within the 100% ± 10% isokinetic requirement for Method 5 sampling.

### 4.2.3 Metals on Particulates

Results of trace metals analysis of particulate filters and probe washes by ICAP are shown in Table 4-3, which combines individual analytical results for filters and probe washes for each Method 5 run. Particulate samples from Run Nos. 3, 5A, and 6A were submitted for dioxin analyses and were not analyzed for trace metals by ICAP; they are not included in Table 4-3. The less than (<) values shown in Table 4-3 are based on the limit of quantification (LOQ) for a specific metal multiplied by the dilution factor. The LOQs for each compound, listed in Table 4-4, represent the lowest

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[4] The McIlvaine Fabric Filter Manual. The McIlvaine Company, 1976. Chapter 9, pp. 157.91-157.93.

## TABLE 4-3. TRACE METALS ANALYSIS OF PARTICULATE FILTERS AND PROBE WASHES

| Metal | Concentration, μg/g | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | Run No. 1A[a] | Run No. 1B | Run No. 2 | Run No. 4 | Run No. 5B | Run No. 6B | Run No. 7A[a] | Run No. 7B | Run No. 8 |
| Aluminum | <3,600 | <4,200 | 6,400 | 2,800 | 6,500 | 6,700 | 6,700 | 6,600 | 5,400 |
| Boron | <240 | <330 | <580 | <610 | <650 | <590 | 1,500 | 1,300 | 870 |
| Barium | <590 | <800 | <690 | <930 | <880 | <690 | <580 | <490 | <580 |
| Beryllium | 15 | 16 | 20 | 18 | 18 | 13 | <11 | 10 | 15 |
| Calcium | 210,000 | 200,000 | 220,000 | 170,000 | 180,000 | 200,000 | 190,000 | 160,000 | 170,000 |
| Cadmium | <77 | <110 | <91 | <120 | <120 | <90 | <75 | 64 | <110 |
| Cobalt | <130 | <160 | 160 | 200 | <210 | <140 | <100 | <170 | 220 |
| Chromium | 2,700 | 3,300 | 1,900 | 2,300 | 2,700 | 2,200 | 1,800 | 2,800 | 2,400 |
| Copper | <630 | <570 | 570 | 740 | 690 | <430 | <310 | <320 | 540 |
| Iron | 7,700 | 5,500 | 5,200 | 10,000 | 7,300 | 5,800 | 3,800 | 23,000 | 17,000 |
| Magnesium | 140,000 | 130,000 | 170,000 | 120,000 | 120,000 | 140,000 | 130,000 | 110,000 | 120,000 |
| Manganese | 250 | 250 | 300 | 290 | 330 | 260 | 220 | 320 | 300 |
| Molybdenum | <180 | <250 | <210 | <290 | <270 | <210 | <180 | <170 | 180 |
| Sodium | <4,800 | <16,000 | <240,000 | <270,000 | <290,000 | <4,300 | <86,000 | <6,100 | <230,000 |
| Nickel | 810 | <640 | 950 | 1,300 | 1,200 | <700 | 610 | 2,630 | 2,500 |
| Lead | <1,300 | <1,700 | <1,500 | <2,000 | <2,400 | 1,700 | <1,200 | 1,200 | 1,500 |
| Tin | <15,000 | <4,400 | <9,900 | <10,000 | <7,800 | <20,000 | <4,800 | <11,000 | <4,900 |
| Titanium | 160 | 170 | 270 | 1,300 | 2,500 | 2,700 | 1,100 | 2,500 | 2,100 |
| Vanadium | 1,100 | 1,400 | 1,800 | 1,400 | 1,400 | 1,100 | 1,000 | 620 | 900 |
| Zinc | 98 | <64 | 1,000 | 1,500 | 1,200 | 1,100 | 1,200 | 1,200 | 1,200 |

[a] Probe wash part of result is average of duplicate analyses.

TABLE 4-4. TYPICAL LOQ's FOR ICAP METAL ANALYSIS

| Metal | LOQ[a] |
|---|---|
| Aluminum | 0.50 |
| Boron | 0.050 |
| Barium | 0.130 |
| Beryllium | 0.005 |
| Calcium | 0.100 |
| Cadmium | 0.020 |
| Cobalt | 0.020 |
| Chromium | 0.030 |
| Copper | 0.060 |
| Iron | 0.033 |
| Magnesium | 0.055 |
| Manganese | 0.005 |
| Molybdenum | 0.04 |
| Sodium | 25.4 |
| Nickel | 0.072 |
| Lead | 0.300 |
| Tin | 5.60 |
| Titanium | 0.020 |
| Vanadium | 0.037 |
| Zinc | 0.005 |

[a] No specific units; units factor of solid or liquid sample (μg/g or μg/L), and dilution factor.

value detected by the ICAP instrument with 95% confidence. Usually, the ICAP instrument can detect metal concentrations lower than the LOQ, but with much less confidence in the accuracy. To obtain the LOQ for a specific metal, the LOQ is miltiplied by the dilution factor for the specific sample.

Metals of highest concentration in the stack gas particulate emissions include calcium (160,000 µg/g to 220,000 µg/g), sodium (4,300 µg/g to 290,000 µg/g), magnesium (110,000 µg/g to 170,000 µg/g), iron (3,800 µg/g to 23,000 µg/g), tin (4,400 µg/g to 20,000 µg/g), and aluminum (2,800 µg/g to 6,700 µg/g).

Mass emission rates for metals, shown in Table 4-5, were calculated using the following equation:

$$M_m = M_s \cdot C_m \cdot \frac{1}{1,000} \qquad (1)$$

where $M_m$ = mass emission rate of trace metal, g/hr
$M_s$ = mass rate of particulates from stack (Table 4-2), kg/hr
$C_m$ = concentration of trace metal (Table 4-3), µg/g
$\frac{1}{1,000}$ = conversion factor to correct units

Metals having highest emission rates include calcium (120 g/hr to 240 g/hr), sodium (<4.5 g/hr to 320 g/hr), magnesium (88 g/hr to 170 g/hr), iron (4.2 g/hr to 31 g/hr), tin (<3.3 g/hr to <20 g/hr), aluminum (2.1 g/hr to 8.8 g/hr), chromium (1.7 g/hr to 3.7 g/hr), lead (<1.3 g/hr to 2.1 g/hr), and vanadium (0.83 g/hr to 1.8 g/hr).

## TABLE 4-5. MASS EMISSION RATE FOR TRACE METALS ON EPA METHOD 5 FILTERS

| | Emission rate, g/hr | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| Metal | Run No. 1A | Run No. 1B | Run No. 2 | Run No. 4 | Run No. 5B | Run No. 6B | Run No. 7A | Run No. 7B | Run No. 8 |
| Aluminum | <4.1 | <3.2 | 6.4 | 2.1 | 5.1 | 6.9 | 7.6 | 8.8 | 7.5 |
| Boron | <0.27 | <0.25 | <0.58 | <0.46 | <0.51 | <0.61 | 1.7 | 1.7 | 1.2 |
| Barium | <0.67 | <0.61 | <0.69 | <0.70 | <0.68 | <0.71 | 0.65 | <0.66 | 0.82 |
| Beryllium | 0.02 | 0.01 | 0.02 | 0.01 | 0.01 | 0.01 | 0.01 | 0.01 | 0.02 |
| Calcium | 230 | 160 | 220 | 120 | 140 | 210 | 210 | 210 | 240 |
| Cadmium | <0.09 | <0.08 | <0.09 | <0.09 | <0.09 | <0.09 | <0.09 | <0.09 | 0.15 |
| Cobalt | <0.15 | <0.12 | 0.16 | 0.15 | <0.17 | <0.15 | <0.12 | <0.22 | 0.31 |
| Chromium | 3.1 | 2.5 | 1.9 | 1.7 | 2.1 | 2.2 | 2.0 | 3.7 | 3.4 |
| Copper | <0.72 | <0.43 | 0.57 | 0.55 | 0.54 | <0.44 | <0.35 | <0.43 | 0.77 |
| Iron | 8.8 | 4.2 | 5.2 | 7.5 | 5.7 | 6.0 | 4.3 | 31 | 25 |
| Magnesium | 160 | 100 | 170 | 88 | 96 | 140 | 140 | 150 | 170 |
| Manganese | 0.29 | 0.19 | 0.30 | 0.22 | 0.25 | 0.27 | 0.25 | 0.43 | 0.43 |
| Molybdenum | <0.21 | <0.19 | <0.21 | <0.21 | <0.21 | <0.22 | <0.20 | <0.23 | 0.26 |
| Sodium | <5.5 | <12 | <240 | <200 | <220 | <4.5 | <97 | <8.3 | 320 |
| Nickel | 0.92 | <0.48 | 0.95 | 0.98 | 0.93 | <0.72 | 0.70 | 3.5 | 3.5 |
| Lead | <1.4 | <1.3 | <1.5 | <1.5 | <1.8 | 1.8 | <1.4 | 1.7 | 2.1 |
| Tin | <17 | <3.3 | <10 | <7.6 | <6.1 | <20 | <5.4 | <15 | <6.9 |
| Titanium | 0.19 | 0.13 | 0.27 | 0.99 | 2.0 | 2.8 | 1.2 | 3.3 | 3.0 |
| Vanadium | 1.3 | 1.1 | 1.8 | 1.1 | 1.1 | 1.1 | 1.3 | 0.83 | 1.3 |
| Zinc | 0.11 | <0.05 | 1.0 | 1.1 | 0.97 | 1.2 | 1.3 | 1.6 | 1.6 |

### 4.2.4 Dioxins on Particulates

Results of dioxin analyses of baghouse dust samples from Runs 3 and 5 and EPA Method 5 particulate filters from Runs 3, 5 and 6A are shown in Table 4-6. All baghouse dust and Method 5 filter samples were below detection limits which are listed in Table 4-6. Detection limits for Method 5 filters are also given in ng/filter. These values were converted to stack gas concentration and expressed in ng/dscm. Acetone probe washes were not sent for dioxin analyses; however, since filter concentrations were not detected it is believed that dioxin concentrations in probe washes would also be below the detection limit.

### 4.2.5 Continuous Monitoring of Carbon Monoxide, Sulfur Dioxides, Nitrogen Oxides, Oxygen, and Total Hydrocarbons

Results of daily continuous monitoring results for carbon monoxide (CO), sulfur dioxide ($SO_2$), nitrogen oxides ($NO_x$), oxygen ($O_2$), and total hydrocarbons (THC) are summarized in Table 4-7. Figures A-1 through A-8 in Appendix A illustrate the 15-minute average emissions for CO, $SO_2$, and $NO_x$ as well as indicate when sampling for particulates and VOST was conducted. The following paragraphs discuss the continuous monitoring results.

Carbon monoxide emissions provide an indication of incomplete combustion in the lime kiln. When the kiln was stable, emission levels remained below 200 ppm. However, process fluctuations or changes in kiln variables caused a momentary or even lengthy excursion in CO emissions to levels greater than 1,000 ppm (0.1%), even during baseline conditions. Emissions of carbon monoxide (CO) ranged from 0 ppm to 5,000 ppm and as indicated by the high standard deviation values fluctuated widely during both baseline and waste fuel conditions. As shown in Figures A-1 through A-8 in Appendix A, particulate and organic stack samples were taken at both high and low CO emission rates.

## TABLE 4-6. DIOXIN RESULTS AT ROCKWELL LIME

| | Baghouse Dust Samples | | | | EPA Method 5 Filters | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Run 3 | | Run 5[a] | | Run 3 | | | Run 5[a] | | | Run 6[a] | | |
| | | | | | | Detection limit | | | Detection limit | | | Detection limit | |
| Dioxin isomer | Sample concen-tration, ppb | Detection limit, ppb | Sample concen-tration, ppb | Detection limit, ppb | Sample concen-tration, ppb | Sample, ng/filter | Stack[b], ng/dscm | Sample concen-tration, ppb | Sample, ng/filter | Stack[b], ng/dscm | Sample concen-tration, ppb | Sample, ng/filter | Stack[b], ng/dscm |
| Tetra | ND[c] | 0.0050 | ND[c] | 0.0090 | ND[c] | 0.041 | 0.034 | ND[c] | 0.51 | 0.45 | ND[c] | 0.067 | 0.057 |
| Penta | ND[c] | 0.014 | ND[c] | 0.024 | ND[c] | 0.13 | 0.11 | ND[c] | 0.16 | 0.14 | ND[c] | 0.16 | 0.14 |
| Hexa | ND[c] | 0.012 | ND[c] | 0.017 | ND[c] | 0.11 | 0.092 | ND[c] | 0.098 | 0.086 | ND[c] | 0.11 | 0.094 |
| Hepta | ND[c] | 0.016 | ND[c] | 0.030 | ND[c] | 0.16 | 0.13 | ND[c] | 0.19 | 0.17 | ND[c] | 0.25 | 0.21 |
| Octa | ND[c] | 0.023 | ND[c] | 0.25 | ND[c] | 1.5 | 1.3 | ND[c] | 1.5 | 1.3 | ND[c] | 2.3 | 2.0 |

[a] Average of split sample.

[b] $\text{ng/dscm} = \frac{\text{ng}}{\text{filter}} \times \frac{1}{\text{dscm sampled}}$

[d] ND= Not detected; below detection limit.

# TABLE 4-7. SUMMARY OF CONTINUOUS MONITORING RESULTS

| | Parameter | | | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | CO, ppm | | | | SO$_2$, ppm | | | | NO$_x$, ppm | | | | O$_2$, % | | | | THC, ppm | | | |
| Day | n[a] | Range | $\bar{X}$[b] | s.d.[c] | n | Range | $\bar{X}$ | s.d. | n | Range | $\bar{X}$ | s.d. | n | Range | $\bar{X}$ | s.d. | n | Range | $\bar{X}$ | s.d. |
| 1 | 25 | 10-4,000 | 1,034 | 1,064 | NA[d] | | | | 22 | 306-379 | 348 | 23 | NA[d] | | | | 12 | 6.7-12.7 | 8.4 | 1.9 |
| 2 | 13 | 20-199 | 45 | 51 | 4 | 123-683 | 528 | 271 | 5 | 326-378 | 345 | 21 | 5 | 7.0-9.3 | 8.2 | 0.9 | NA[e] | | | |
| 3 | 30 | 18-4,900 | 200 | 888 | 21 | 492-622 | 557 | 59 | 21 | 417-463 | 437 | 13 | 21 | 5.8-6.3 | 6.1 | 0.2 | NA[f] | | | |
| 4 | 34 | 10-147 | 32 | 29 | 34 | 196-639 | 492 | 103 | 34 | 307-478 | 439 | 42 | 34 | 5.6-8.6 | 6.2 | 0.5 | 25 | 2.5-10.0 | 3.9 | 1.6 |
| 5 | 38 | 18-1,600 | 224 | 457 | 33 | 433-701 | 540 | 65 | 33 | 425-552 | 507 | 36 | 33 | 4.9-6.6 | 5.6 | 0.4 | 37 | 1.5-6.0 | 3.0 | 0.9 |
| 6 | 34 | 20-3,843 | 557 | 996 | 36 | 370-2,067 | 637 | 274 | 36 | 425-510 | 469 | 28 | 35 | 4.0-6.9 | 5.0 | 0.7 | 27 | 2.0-5.0 | 3.5 | 0.9 |
| 7 | 38 | 39-12,910 | 1,060 | 2,326 | 35 | 183-985 | 650 | 224 | 35 | 346-479 | 439 | 29 | 34 | 4.2-6.2 | 5.3 | 0.7 | 32 | 2.3-5.6 | 3.8 | 1.0 |
| 8 | 22 | 30-4,537 | 1,357 | 1,514 | 21 | 269-1,924 | 672 | 420 | 21 | 288-418 | 258 | 32 | 21 | 1.8-10.0 | 6.0 | 2.3 | 22 | 2.8-6.0 | 3.6 | 0.8 |

[a] n = Number of readings; one reading is integrated 15-minute average from continuous monitoring.

[b] $\bar{X}$ = Mean value.

[c] s.d. = Standard deviation value.

[d] NA = Not analyzed continuously on Day 1.

[e] NA = Not analyzed continuously on Day 2 due to set-up problems.

[f] NA = Not analyzed continuously on Day 3 due to leak in gas cylinder.

Sulfur dioxide ($SO_2$) concentrations ranged from 100 ppm to 2,000 ppm over the entire test period. No continuous $SO_2$ data was collected on Day 1, instead two EPA Method 6 runs were conducted. Continuous data was obtained only for the last hour of Run No. 2. All other runs (Run Nos. 2 through 8) have continuous $SO_2$ data for the duration of the Day. The average baseline $SO_2$ concentration was 553 ppm ± 46 ppm (95% confidence limit), and the average waste fuel condition $SO_2$ concentration was 596 ppm ± 38 ppm.

Nitrogen oxides ($NO_x$) concentrations ranged from approximately 290 ppm to 550 ppm. Average baseline $NO_x$ concentration was 386 ppm ± 100 ppm (95% confidence limit); the average waste fuel condition $NO_x$ concentration was 446 ppm ± 129 ppm. Continuous monitoring was conducted on Days 1 through 8; however, data was collected for only one hour on Day 2 due to instrument start-up problems. Figures A-1 through A-8 in Appendix A show 15-minute integrated $NO_x$ concentrations for each run.

Percent oxygen ($O_2$), monitored on Days 2 through 8, ranged from 1.8% to 10.0% for all runs. The average $O_2$ concentration during baseline condition was 6.5% ± 1.9% (95% confidence limit); the average waste fuel $O_2$ concentration was 5.6% ± 3.0%. Oxygen monitoring conducted by Rockwell Lime Company at the kiln exit had excellent comparison with the MRC $O_2$ monitor during periodic checks. In addition, Fyrite $O_2$ measurements taken during Method 5 runs compared well with the continuous $O_2$ monitoring results. Figures A-9 through A-17 in Appendix A shows the 15-minute integrated averages for $O_2$ monitoring for each run.

Total hydrocarbons (THC) measured continuously during Days 1 and 4 through 8 indicate combustion efficiency or the emission rate of any unburned hydrocarbons. No THC was collected on Day 2 due to sampling problems or on Day 3 due to a leak in the hydrogen gas cylinder of the analytical instrument. THC concentrations ranged from 1.5 ppm to 12.7 ppm. The average THC baseline concentration (one day only) was 8.4 ppm ± 3.8 ppm (95% confidence limit); the average THC concentration during the waste fuel condition was 3.5 ppm ± 2 ppm. Figures A-9 through A-17 in Appendix A shows the 15-minute average values for THC for each run. The low THC concentration shown in the data indicate that, although THC concentration during waste fuel conditions was less than baseline, under either condition good combustion was maintained.

### 4.2.6 $SO_2$ and $NO_x$ Noncontinuous Results

Sampling for $SO_2$ by EPA Method 6 was conducted on Day 1 (two samples) and Day 2 when no continuous $SO_2$ monitoring was performed, and on Days 3 and 7 to compare with $SO_2$ continuous monitoring data. Results of $SO_2$ sampling are shown in Table 4-8. Results for sample Nos. 3A, 7A, and 7B compare very well with continuous $SO_2$ results obtained for the sampling period (averaging 7% relative difference).

Sampling for $NO_x$ by EPA Method 7 was conducted only on Day 3 (four samples) for comparison with continuous $NO_x$ monitoring. Results of $NO_x$ sampling also are presented in Table 4-8.

### 4.2.7 POHC Emissions ($W_{out}$)

A total of 45 runs was made over the eight sampling days. Six volatile organic sample train (VOST) runs were made each day, except on Day 3 when one run was voided due to leak check problems and on Day 8 when the last two runs were cancelled because the waste fuel supply was exhausted. The total nanograms (ng) of each POHC collected, shown in Table 4-9, were calculated by adding the front and back tube masses. VOST tube blanks (lab, trip, and field) are shown in Table 4-10 for each POHC. Mass values shown in Table 4-9 do not include the blank values because (1) not subtracting the blanks from the mass collected represents

## TABLE 4-8. $SO_2$ and $NO_x$ NONCONTINUOUS SAMPLING RESULTS FOR STACK GAS

| Run No. | $SO_2$[a] emission rate kg/hr | $SO_2$[a] emission rate lb/hr | $SO_2$ concentration mg/m$^3$ | $SO_2$ concentration ppm | $NO_x$[b] emission rate kg/hr | $NO_x$[b] emission rate lb/hr | $NO_x$ concentration mg/m$^3$ | $NO_x$ concentration ppm | Continuous $SO_2$/$NO_x$ results, ppm |
|---|---|---|---|---|---|---|---|---|---|
| 1A | 91 | 201 | 2,960 | 1,145 | | | | | - |
| 1B | 98 | 215 | 3,160 | 1,210 | | | | | - |
| 2A | 44 | 96 | 1,310 | 500 | | | | | - |
| 3A | 39 | 85 | 1,400 | 535 | | | | | 587 |
| 7A | 45 | 99 | 1,650 | 628 | | | | | 589 |
| 7B | 43 | 95 | 1,580 | 603 | | | | | 546 |
| 3A | | | | | 55 | 121 | 1,995 | 1,050 | 440 |
| 3B | | | | | 60 | 131 | 2,160 | 1,140 | 418 |
| 3C | | | | | 64 | 141 | 2,330 | 1,230 | 434 |
| 3D | | | | | 65 | 143 | 2,350 | 1,240 | 463 |

[a] Run Nos. 1A, 1B, 2A, 3A, 7A, and 7B for $SO_2$ results only.

[b] Run Nos. 3A, 3B, 3C, and 3D for $NO_x$ results only.

a worst case or maximum value when calculating destruction efficiency run, and (2) approximately 50% of the mass values were less than the blank values for the six POHCs.

The mass values shown in Table 4-9 and the VOST sample volumes and stack gas rates were used to calculate the POHC emission rates shown in Table 4-11 ($W_{out}$ values) using the following equation:

$$W_{out} = \frac{M_t}{Vol_s} \cdot V_s \cdot \frac{1}{10^9} \qquad (2)$$

## TABLE 4-9. TOTAL MASS ON VOST TUBES

| Run No. | Front and back tube mass, g (assume blank = 0) | | | | | |
|---|---|---|---|---|---|---|
| | $MeCl_2$ | MEK | $CH_3CCl_3$ | TCE | Perc | Toluene |
| 1A | 1.91 | 23.54 | 8.31 | 200.5 | $4.5^a$ | $90.3^a$ |
| 1B | $1.63^a$ | $3.57^a$ | $6.70^a$ | $5.89^a$ | $3.59^a$ | $47.0^a$ |
| 1C | 1.82 | 13.48 | 86.04 | 6.29 | $7.81^b$ | $19.4^b$ |
| 1D | 1.32 | 10.10 | 11.52 | 4.60 | 10.99 | 102.8 |
| 1E | 1.54 | 5.96 | 16.06 | 4.59 | 12.74 | 136.6 |
| 1F | 0.82 | 6.43 | 8.70 | 2.87 | 9.55 | 89.28 |
| 2A | 6.85 | 25.30 | 6.06 | 7.69 | 12.39 | 59.47 |
| 2B | 8.81 | 45.00 | 36.40 | 20.63 | 14.03 | 47.93 |
| 2C | 11.18 | 67.10 | 18.87 | 3.08 | 6.23 | 17.19 |
| 2D | 0.71 | 24.70 | 8.27 | 3.35 | 7.59 | 18.42 |
| 2E | 1.26 | 25.70 | 6.24 | 5.08 | 7.51 | 38.60 |
| 2F | $1.09^b$ | $22.40^b$ | $5.84^b$ | $19.40^b$ | $11.30^b$ | $49.10^b$ |
| 3A | 2.66 | 22.66 | 8.39 | 9.07 | 9.22 | 45.30 |
| 3B | 7.51 | 44.30 | 35.30 | 20.23 | 24.30 | 86.70 |
| 3C | 0.67 | 27.50 | 6.23 | 2.88 | 6.01 | 30.90 |
| 3D | 0.83 | 26.00 | 10.60 | 8.51 | 10.63 | 52.60 |
| 3E | 0.57 | 32.43 | 1.25 | 4.07 | 8.90 | 64.63 |
| Average of baseline Run Nos. 1 through 3 | 3.01 | 25.07 | 16.87 | 19.34 | 9.84 | 58.60 |
| 4A | 4.78 | 15.13 | 7.58 | $4.84^c$ | 7.23 | 43.98 |
| 4B | $3.64^c$ | 36.11 | 14.66 | $5.46^c$ | $5.28^a$ | 44.22 |
| 4C | $0.55^a$ | $19.80^a$ | $6.44^a$ | $3.52^a$ | $4.60^a$ | $33.20^a$ |
| 4D | 10.38 | 49.44 | 25.30 | 3.45 | $3.66^b$ | 53.60 |
| 4E | 30.53 | 25.26 | 49.50 | $5.05^c$ | $9.09^c$ | 56.78 |
| 4F | $8.81^b$ | $4.39^b$ | $7.44^b$ | $1.61^b$ | $3.36^b$ | $4.33^b$ |
| 5A | $1,066.4^b$ | $17.0^b$ | $39.0^b$ | $14.6^b$ | $0.18^b$ | $0.82^b$ |
| 5B | $1.39^b$ | $5.90^b$ | $2.14^b$ | $3.06^b$ | $1.42^b$ | $4.97^b$ |
| 5C | $5.73^d$ | 74.90 | 5.00 | 5.54 | 4.05 | 68.19 |
| 5D | 1.55 | 19.85 | 4.29 | 8.65 | 4.68 | 76.43 |
| 5E | 17.16 | 43.20 | 14.50 | 12.32 | 10.65 | 156.99 |
| 5F | 54.97 | 31.20 | 8.21 | 11.38 | 5.31 | 101.91 |
| 6A | 1.43 | 41.80 | 4.03 | 5.26 | 3.88 | 23.96 |
| 6B | $0.55^a$ | $59.0^a$ | $2.76^a$ | $4.04^a$ | $3.26^a$ | $31.90^a$ |
| 6C | $11.00^b$ | $17.6^b$ | $106.5^b$ | $32.60^b$ | $4.65^b$ | $30.40^b$ |
| 6D | 1.34 | 28.02 | 7.87 | 6.61 | 5.24 | 139.37 |
| 6E | $3.97^d$ | $13.8^d$ | $22.09^d$ | 7.77 | 14.34 | 134.78 |
| 6F | 2.66 | 39.50 | 27.95 | 21.16 | 14.65 | 149.49 |

(continued)

## TABLE 4-9 (continued)

| Run No. | Front and back tube mass, g (assume blank = 0) | | | | | |
|---|---|---|---|---|---|---|
| | $MeCl_2$ | MEK | $CH_3CCl_3$ | TCE | Perc | Toluene |
| 7A | 1.42 | 29.72 | 7.48 | 2.46 | 3.69 | 91.01 |
| 7B | 2.63 | 53.10 | 6.81 | 5.05 | 35.70 | 81.18 |
| 7C | 1.71 | 103.30 | 32.72 | 5.44 | 19.95 | 120.38 |
| 7D | 6.99 | 88.30 | 9.07 | 6.32 | 16.67 | 299.57 |
| 7E | 5.67 | 13.0[d] | 71.90 | 30.60 | 19.59 | 416.54 |
| 7F | 19.35 | 32.24 | 21.24 | 5.47 | 16.02 | 167.40 |
| 8A | 6.16[b] | 15.60[b] | 22.3[b] | 17.85 | 29.50 | 272.40 |
| 8B | 11.67 | 58.20 | 40.70 | 14.59 | 44.60 | 154.70 |
| 8C | 4.80[d] | 17.50[d] | 50.10 | 27.77 | 67.39 | 224.60 |
| 8D | 2.94 | 97.60 | 81.60 | 40.61 | 40.80 | 167.00 |
| Average of waste fuel Run Nos. 4 through 8 | 8.29[e] | 37.87 | 24.97 | 11.18 | 14.27 | 112.50 |

---

[a] NAI = back tube not analyzed.

[b] NAI = front tube not analyzed.

[c] Back tube not detected.

[d] Front tube mass not detected or less than blank.

[e] Average does not include Run No. 5A (1,006 ng).

$$W_{out} = \frac{M_t}{Vol_s} \cdot V_s \cdot \frac{1}{10^9} \qquad (2)$$

where $W_{out}$ = emission rate, g/min

$M_t$ = mass on tubes, ng, (Table 4-9)

$Vol_s$ = volume of gas sampled from VOST runs (dry $m^3$)

$V_s$ = stack gas rate from Method 5 runs (dry $m^3$/min)

$\frac{1}{10^9}$ = conversion factor for g to ng

The emission rates in Table 4-11 will be used in Section 5 to calculate destruction and removal efficiencies (DREs) for a given POHC.

## TABLE 4-.10 VOST TUBE BLANK MASS VALUES

| | Mass, ng | | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Laboratory | | | | | | | Trip | | | | Field | | | | | | | | Average of field blanks |
| | | | | | | | | | | | | A[a] | | B[a] | | C | | D | | |
| Analyte | A[a] | B[a] | C[a] | D | E | F | G | A[a] | B[a] | C | D | fr[b] | bk[c] | fr | bk | fr | bk | fr | bk | |
| Methylene chloride | - | - | - | 3.85 | 1.85 | 0.71 | 4.85 | - | - | 0.46 | 0.97 | - | - | - | - | 0.62 | 5.31 | 0.43 | 0.31 | 1.67 |
| Methyl ethyl ketone | - | - | - | 5.34 | 1.58 | 4.96 | 10.4 | - | - | 2.40 | 2.78 | - | - | - | - | 4.56 | 6.59 | 3.30 | 2.47 | 4.23 |
| 1,1,1-Trichloroethane | - | - | - | 55.6 | 12.7 | 3.46 | 8.01 | - | - | 1.77 | 8.08 | - | - | - | - | 3.80 | 25.9 | 2.62 | 4.41 | 9.18 |
| Trichloroethylene | 1.4 | 1.3 | 2.4 | 2.60 | 7.31 | 0.98 | 4.53 | 1.0 | 3.0 | 1.25 | 2.46 | 1.2 | 1.4 | 1.2 | 1.5 | 0.92 | 17.6 | 1.96 | 2.41 | 3.52 |
| Tetrachloroethylene | 1.4 | 1.4 | 1.1 | 1.72 | 1.45 | 2.70 | 5.32 | 0.85 | 2.6 | 1.22 | 5.40 | 2.9 | 3.0 | 2.8 | 2.9 | 6.54 | 10.5 | 4.59 | 5.33 | 4.82 |
| Toluene | - | - | - | 6.05 | 18.1 | 4.59 | 12.7 | - | - | 2.50 | 10.7 | - | - | - | - | 5.95 | 21.2 | 6.39 | 7.29 | 10.21 |

[a] Lab blanks for TCE and Perc analyzed after first trip on 4/15/83 prior to identification of new POHCs.

[b] Front tube blank (fr).

[c] Back tube blank (bk).

TABLE 4-11. TOTAL MASS OUT BASED ON VOST RUNS ($W_{out}$)

| Run No. | Mass out ($W_{out}$), X $10^{-5}$ g/min[a] | | | | | |
|---|---|---|---|---|---|---|
| | $MeCl_2$ | MEK | $CH_3CCl_3$ | TCE | Perc | Toluene |
| 1A | 3.552 | 43.775 | 15.453 | 372.85 | 8.368[b] | 167.92[b] |
| 1B | 3.073[b] | 6.731[b] | 12.632[b] | 11.105[b] | 6.769[b] | 88.615[b] |
| 1C | 3.408 | 25.240 | 161.103 | 11.777 | 14.624[c] | 36.325[c] |
| 1D | 2.364 | 18.089 | 20.632 | 8.238 | 19.682 | 184.109 |
| 1E | 2.739 | 10.600 | 28.563 | 8.163 | 22.659 | 242.950 |
| 1F | 1.463 | 11.476 | 15.527 | 5.122 | 17.044 | 159.340 |
| 2A | 10.133 | 37.424 | 8.964 | 11.375 | 18.328 | 87.969 |
| 2B | 14.299 | 73.040 | 59.081 | 33.485 | 22.756 | 77.795 |
| 2C | 20.169 | 121.052 | 34.043 | 55.565 | 11.239 | 31.012 |
| 2D | 1.272 | 44.261 | 14.819 | 6.003 | 13.609 | 33.008 |
| 2E | 2.344 | 47.818 | 11.610 | 9.452 | 13.973 | 71.820 |
| 2F | 1.987[c] | 40.825[c] | 10.644[c] | 35.357[c] | 20.595[c] | 89.486[c] |
| 3A | 3.955 | 33.696 | 12.476 | 13.487 | 13.710 | 67.362 |
| 3B | 11.739 | 69.247 | 55.179 | 31.622 | 37.984 | 135.524 |
| 3C | 1.040 | 42.695 | 9.672 | 4.471 | 9.331 | 47.973 |
| 3D | 1.181 | 36.981 | 15.088 | 12.104 | 15.120 | 74.816 |
| 3E | 1.000 | 56.908 | 2.193 | 7.142 | 15.618 | 113.412 |
| Average of baseline Run Nos. 1 through 3 | 5.042 | 42.345 | 28.687 | 37.489 | 16.553 | 100.555 |
| 4A | 7.379 | 23.358 | 11.702 | 7.472 | 11.162 | 67.896 |
| 4B | 5.529 | 54.850 | 22.268 | 8.294 | 8.020[b] | 67.169 |
| 4C | 0.952[b] | 34.262[b] | 11.144 | 6.091[b] | 7.960 | 57.449[b] |
| 4D | 17.266 | 82.238 | 42.084 | 5.739 | 6.088 | 89.157 |
| 4E | 53.815 | 44.525 | 87.253 | 8.902 | 16.023 | 100.085 |
| 4F | 14.253 | 7.102 | 12.036 | 2.605 | 5.436 | 7.005 |
| 5A | 1,861.2[c] | 29.671[c] | 68.069[c] | 25.482[c] | 0.314[c] | 1.431[c] |
| 5B | 2.260[c] | 9.591 | 3.479[c] | 4.974[c] | 2.308[c] | 8.063[c] |
| 5C | 7.248 | 94.740 | 6.324 | 7.007 | 5.123 | 86.253 |
| 5D | 2.304 | 44.361 | 6.375 | 12.855 | 6.955 | 113.586 |
| 5E | 27.113 | 68.256 | 22.910 | 19.466 | 16.827 | 248.044 |
| 5F | 79.761 | 45.321 | 11.926 | 16.530 | 7.713 | 148.032 |

(continued)

TABLE 4-11 (continued)

| Run No. | Mass out ($W_{out}$), X $10^{-5}$ g/min[a] | | | | | |
|---|---|---|---|---|---|---|
| | $MeCl_2$ | MEK | $CH_3CCl_3$ | TCE | Perc | Toluene |
| 6A | 1.949 | 56.986 | 5.494 | 7.171 | 5.289 | 32.665 |
| 6B | 0.907[b] | 97.309[b] | 4.552[b] | 6.663[b] | 5.377[b] | 52.613[b] |
| 6C | 16.368[c] | 26.189[c] | 158.473[c] | 48.509[c] | 6.919[c] | 45.236[c] |
| 6D | 1.795 | 37.548 | 10.546 | 8.858 | 7.022 | 186.764 |
| 6E | 7.230 | 25.133 | 40.231 | 14.151 | 26.117 | 245.466 |
| 6F | 4.717 | 7.005 | 49.564 | 37.523 | 25.979 | 265.092 |
| 7A | 2.362 | 49.446 | 12.445 | 4.093 | 6.139 | 151.416 |
| 7B | 6.032 | 121.783 | 15.618 | 11.582 | 81.876 | 186.183 |
| 7C | 3.120 | 188.474 | 59.699 | 9.925 | 36.400 | 219.638 |
| 7D | 11.830 | 149.440 | 15.350 | 10.696 | 28.213 | 506.996 |
| 7E | 10.269 | 23.544 | 130.216 | 55.419 | 35.479 | 754.383 |
| 7F | 49.463 | 82.414 | 54.295 | 13.983 | 40.951 | 427.916 |
| 8A | 11.409[c] | 28.892[c] | 41.301[c] | 33.059 | 54.636 | 504.501 |
| 8B | 20.909 | 104.275 | 72.921 | 26.140 | 79.908 | 277.171 |
| 8C | 7.731 | 28.188 | 80.699 | 44.731 | 108.548 | 361.744 |
| 8D | 6.160 | 204.505 | 170.980 | 85.092 | 85.499 | 349.922 |
| Average of waste fuel Run Nos. 4 through 8 | 14.079 | 63.072 | 44.106 | 19.715 | 26.010 | 201.230 |

[a] $W_{out}$, g/min = $\frac{\text{mass on tubes (ng)}}{\text{volume sampled (dm}^3\text{)}}$ x stack rate $(\frac{dm^3}{min})$ x $\frac{g}{10^9\ ng}$, assumes blank = 0.

[b] Front tube only.

[c] Back tube only.

## 4.3 PROCESS SAMPLE RESULTS

### 4.3.1 Waste Fuel ($W_{in}$)

Results of waste fuel analyses for chlorine, sulfur, PCB, heat value, specific gravity, and feed rate are shown in Table 4-12. Chlorine content averaged 3.07%, which includes spikes of trichloroethylene and tetrachloroethylene that were added to the waste fuel to improve the evaluation of their destruction. Feed

## TABLE 4-12. WASTE FUEL CONDITIONS

| Run No.[a] | Date | Chlorine content, % (vol.) | Sulfur content, % | PCB concentration, ppm[b] | Heat value, Btu/lb | Specific gravity, g/cc | Feed rate | | Mass rate | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | L/min | gal/min | g/min | lb/min |
| 4 | 05/02/83 | 3.14 | 0.10 | 1.0 | 12,301 | 1.031 | 2.42 | 0.64[c] | 2,498 | 5.50 |
| | | | | | | | 3.40 | 0.89[d] | 3,473 | 7.65 |
| 5 | 05/03/83 | 2.66 | 0.08 | 1.0 | 12,084 | 1.042 | 4.58 | 1.21 | 4,772 | 10.51 |
| 6[e] | 05/04/83 | 3.04 | 0.065 | 1.0 | 12,267 | 1.035 | 7.76 | 2.05 | 8,023 | 17.67 |
| 7 | 05/05/83 | 3.05 | 0.06 | 1.0 | 13,612 | 0.986 | 2.95 | 0.78[f] | 2,911 | 6.41 |
| | | | | | | | 10.98 | 2.90[g] | 10,823 | 23.84 |
| 8 | 05/06/83 | 3.51 | 0.11 | 1.0 | 14,064 | 0.971 | 10.40 | 2.88 | 10,585 | 23.32 |

[a] No waste fuel burned during Run Nos. 1, 2, and 3.

[b] Analyses performed on one waste fuel sample prior to field test.

[c] Between 0800 and 1305 hours.

[d] Between 1305 and 1600 hours.

[e] Average of duplicate samples.

[f] Between 0800 and 1100 hours.

[g] Between 1100 and 1630 hours.

rates ranged from 2,500 g/min (5.50 lb/min) during the first waste fuel test days to over 10,500 g/min (23.3 lb/min) at the end of the test. Analysis for PCB was conducted prior to the test burn to meet test burn permit requirements, which allowed a maximum PCB concentration of 10 ppm in the waste fuel.

Two waste fuel samples from Days 4 and 7 were analyzed for the twenty major components using capillary GC/MS, and results are shown in Table 4-13. As noted on the list, toluene, 2-ethoxyethyl acetate, and xylene are in the highest concentration. The sums of the concentrations listed represent only 35% to 40% of the total sample weight. About one-half of the sample was composed of a sludge-like precipitate, which was largely insoluble in organic solvents and could not be analyzed by GC/MS, thus accounting for the relatively small percentage of the sample weight analyzed. In general, these results agree well with GC analyses of the waste fuel conducted by Hydrite Chemical Company prior to the test burn.

For Run 4, 1,1,1-trichloroethane, trichloroethylene, and tetrachloroethylene are the only chlorinated compounds among the top twenty listed in Table 4-13. These chlorinated compounds represent 4.5 wt% of the top twenty compounds listed in Table 4-13; this compares well with the 5.3% (by weight) total chlorine listed in Table 4-12 (3.07% by volume). The remaining chlorine (0.8% by weight) is most likely attributable to low concentration organic chlorinated species in the waste fuel and inorganic chlorides in the sludgy material.

The concentration of POHCs in the waste fuel is shown in Table 4-14. All of the POHCs were relatively constant in concentration during the test. Results for methylene chloride should be reviewed with caution because blank samples showed contamination of methylene chloride, a common organic solvent used in analytical extractions.

TABLE 4-13. RESULTS OF CAPILLARY GC/MS ANALYSIS OF MAJOR COMPONENTS OF WASTE FUELS NOS. 4 and 7

| Waste fuel components | Concentration (wt %) Fuel No. 4 | Fuel No. 4 (duplicate) | Fuel No. 7 | Fuel No. 7 (duplicate) |
|---|---|---|---|---|
| Acetone | 0.242 | 0.216 | 0.228 | 0.218 |
| Methyl ethyl ketone[a] | 2.48 | 2.47 | 2.99 | 3.34 |
| 1,1,1-Trichloroethane[a] | 0.255 | 0.221 | 0.198 | 0.233 |
| 1-Butanol | 0.325 | 0.316 | 0.350 | 0.390 |
| Trichloroethylene[a] | 1.97 | 1.49 | 2.38 | 1.93 |
| 2-Ethoxyethanol | 0.872 | 0.831 | 0.890 | 0.940 |
| Methyl isobutyl ketone | 1.14 | 0.982 | 1.13 | 1.20 |
| Toluene[a] | 12.0 | 9.94 | 12.6 | 12.4 |
| Tetrachloroethylene[a] | 2.26 | 2.08 | 2.45 | 2.52 |
| Butyl acetone | 0.281 | 0.261 | 0.285 | 0.350 |
| Ethylbenzene | 1.44 | 1.39 | 1.65 | 1.51 |
| Xylene (isomer No. 1) | 4.96 | 4.88 | 5.73 | 5.42 |
| Xylene (isomer No. 2) | 1.46 | 1.40 | 1.64 | 1.56 |
| 2-Butoxyethyanol | 2.06 | 1.92 | 2.11 | 2.03 |
| 2-Ethoxyethyl acetate | 6.07 | 5.74 | 6.56 | 6.18 |
| $C_3$-Benzene (isomer No. 1) | 0.318 | 0.250 | 0.345 | 0.299 |
| $C_3$-Benzene (isomer No. 2) | 0.458 | 0.470 | 0.565 | 0.579 |
| $C_{10}$-Alkane | 0.831 | 0.770 | 0.966 | 0.922 |
| Alkane $>C_8$ | 0.228 | 0.253 | 0.280 | 0.273 |
| Alkane | 0.155 | 0.134 | 0.183 | 0.174 |
| $C_{11}$-Alkane | 1.29 | 1.23 | 1.51 | 1.45 |
| 2-Cyclohexen-1-one or 3,5,5-Trimethyl (isomer) | 0.160 | 0.151 | 0.161 | 0.197 |
| Alkane $>C_6$ | 0.211 | 0.196 | 0.246 | 0.240 |

[a] POHCs (note $MeCl^2$ not on this list).

Mass feed rates ($W_{in}$) for each POHC for each run are included in Table 4-14. The waste fuel feed rates was varied on Days 4 and 7 resulting in two mass rates for each POHc on those days. The mass feed rate ($W_{in}$) values are used in Section 5 to calculate the destruction and removal efficiencies for each POHC.

Table 4-15 shows the concentrations of trace metals in the waste fuel samples. The metals of highest concentration and above the limit of quantification (LOQ) for the ICAP instrument include zinc (250 mg/L to 270 mg/L), lead (130 mg/L to 140 mg/L), iron (30 mg/L to 39 mg/L), boron (19 mg/L to 37 mg/L), cobalt (22 mg/L to 25

TABLE 4-14. CONCENTRATION AND MASS RATE OF POHCs ($W_{in}$)

| Run No. | Waste fuel rate, g/min | POHCs concentration, wt %[a] | | | | | |
|---|---|---|---|---|---|---|---|
| | | $MeCl_2$ | MEK | $CH_3CCl_3$ | TCE | Perc | Toluene |
| 1A-1F | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| 2A-2F | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| 3A-3E | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| 4A-4D | 2,497.5 | 0.101 | 2.48 | 0.238 | 1.73 | 2.17 | 10.97 |
| 4E-4F | 3,473.1 | 0.101 | 2.48 | 0.238 | 1.73 | 2.17 | 10.97 |
| 5A-5F | 4,772.2 | 0.097 | 2.75 | 0.239 | 1.64 | 2.02 | 10.55 |
| 6A-6F | 8,023.1 | 0.106[b] | 2.48[b] | 0.228[b] | 1.78[b] | 2.05[b] | 10.95[b] |
| 7A-7C | 2,911.0 | 0.120 | 3.17 | 0.216 | 2.16 | 2.49 | 12.50 |
| 7D-7F | 10,822.8 | 0.120 | 3.17 | 0.216 | 2.16 | 2.49 | 12.50 |
| 8A-8D | 10,584.7 | 0.116 | 2.59 | 0.282 | 1.89 | 2.56 | 12.90 |
| 1A-1F | 0 | | | | | | |
| 2A-1F | 0 | | | | | | |
| 3A-3E | 0 | | | | | | |
| 4A-4D | 2,497.5 | 2.52 | 61.94 | 5.94 | 43.21 | 54.20 | 273.97 |
| 4E-4F | 3,473.1 | 3.51 | 86.13 | 8.27 | 60.09 | 75.37 | 381.00 |
| 5A-5F | 4,772.2 | 4.63 | 131.24 | 11.41 | 78.26 | 96.40 | 503.47 |
| 6A-6F | 8,023.1 | 8.50 | 198.97 | 18.29 | 142.81 | 164.47 | 878.53 |
| 7A-7C | 2,911.0 | 3.49 | 92.28 | 6.29 | 62.88 | 72.48 | 363.87 |
| 7D-7F | 10,822.8 | 12.99 | 343.08 | 23.38 | 233.77 | 269.49 | 1,352.85 |
| 8A-8D | 10,584.7 | 12.28 | 274.14 | 29.85 | 200.05 | 270.97 | 1,365.42 |

[a] From waste fuel analyses; average of split analyses.

[b] Average of four samples (split of duplicate samples).

[c] $W_{in}$, g/min = (fuel rate, g/min) x (wt %).

mg/L), chromium (11 mg/L to 12 mg/L), and manganese and titanium (2 mg/L to 3 mg/L). Every other metal was below the LOQ for that element. As described in Section 4.3.2, the LOQ values are based on the lowest detectable value (at 95% confidence) times the dilution factor. Typical ICAP LOQ values were listed in Table 4-4.

### 4.3.2 Petroleum Coke Results

Results from the analyses of coke samples are shown in Table 4-16. The coke feed rate is a daily average of hourly readings recorded

TABLE 4-15. TRACE METALS RESULTS FOR WASTE FUEL SAMPLES

| | Concentration, mg/L | | | | |
|---|---|---|---|---|---|
| Metal | Fuel No. 4 | Fuel No. 5 | Fuel No. 6[a] | Fuel No. 7 | Fuel No. 8 |
| Aluminum | <50 | <50 | <50 | <50 | <50 |
| Boron | 19 | 37 | 28 | 28 | 15 |
| Barium | <13 | <13 | <13 | <13 | <13 |
| Beryllium | <0.20 | <0.20 | <0.20 | <0.20 | <0.20 |
| Calcium | <9.6 | <9.6 | <13 | 54 | 59 |
| Cadmium | <1.7 | <1.7 | <1.7 | <1.7 | <1.7 |
| Cobalt | 25 | 23 | 22 | 23 | 23 |
| Chromium | 11 | 12 | 12 | 12 | 11 |
| Copper | <5.9 | <5.9 | <6.0 | <5.9 | 13 |
| Iron | 30 | 34 | 32 | 39 | 33 |
| Magnesium | <0.20 | <0.20 | <0.20 | <0.20 | <0.20 |
| Manganese | 2.2 | 2.2 | 2.1 | 2.5 | 2.3 |
| Molybdenum | <4.0 | <4.0 | <4.0 | <4.0 | <4.0 |
| Sodium | <2,500 | <2,500 | <2,500 | <2,500 | <2,500 |
| Nickel | <7.2 | <7.2 | <7.2 | <7.2 | <7.2 |
| Lead | 130 | 130 | 130 | 140 | 130 |
| Tin | <560 | <560 | <560 | <560 | <560 |
| Titanium | 2.6 | 2.6 | 2.3 | 2.6 | 2.4 |
| Vanadium | <3.7 | <3.7 | <3.7 | <3.7 | <3.7 |
| Zinc | 250 | 250 | 250 | 270 | 260 |

[a] Average of duplicate samples.

by the Rockwell kiln operators. Sulfur content averaged approximately 4%, and Btu content averaged 32,970 Joules/g (14,190 Btu/lb) over the eight-day test period.

Trace metal results for the petroleum coke are shown in Table 4-17.

### 4.3.3 Limestone Feed, Lime Product, and Baghouse Dust Results

Chloride and sulfur analyses and mass rates for limestone feed, lime product, and baghouse dust samples are shown in Table 4-18.

## TABLE 4-16. COKE CONDITIONS AND ANALYTICAL RESULTS

| Run No. | Run condition | Sulfur content, % | Chloride content, % | Ash content, % | Heat value, Btu/lb | Feed rate | |
|---|---|---|---|---|---|---|---|
| | | | | | | kg/hr | lb/hr |
| 1 | Baseline | 3.92 | 0.039 | 2.42 | 15,000 | 1,453[a] | 3,200 |
| 2 | Baseline | 3.95 | 0.038 | 1.03 | 13,810 | 1,453[a] | 3,200 |
| 3 | Baseline | 4.28 | 0.071 | 0.54 | 14,207 | 1,453[a] | 3,200 |
| 4 | Waste fuel | 4.02 | 0.026 | 0.73 | 14,073 | 1,362 | 3,000 |
| 5 | Waste fuel | 4.02 | 0.028 | 0.52 | 13,961 | 1,362 | 3,000 |
| 6[b] | Waste fuel | 4.11 | 0.047 | 0.36 | 14,214 | 1,362 | 3,000 |
| 7 | Waste fuel | 4.17 | 0.039 | 0.30 | 14,155 | 1,476[c]<br>1,226[d] | 3,250[c]<br>2,700[d] |
| 8 | Waste fuel | 4.07 | 0.234 | 0.30 | 14,064 | 1,112 | 2,450 |

[a] Approximate values.

[b] Average of duplicate samples.

[c] Coke feed rate prior to 1130 hours.

[d] Coke feed rate after 1200 hours.

# TABLE 4-17. METAL RESULTS FOR PETROLEUM COKE SAMPLES

| Metal | Concentration, µg/gram | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | Run No. 1 | Run No. 2 | Run No. 3 | Run No. 4 | Run No. 5 | Run No. 6 | Run No. 7 | Run No. 8 |
| Aluminum | 54 | <95 | <98 | <82 | <88 | <100 | <98 | <97 |
| Boron | <2.5 | <10 | <10 | <8.7 | <9.3 | <17 | 20 | <10 |
| Barium | 2.1 | <25 | <25 | <21 | <23 | <26 | <25 | <25 |
| Beryllium | <0.5 | 2.0 | 1.5 | 1.8 | 1.4 | <0.9 | 1.3 | 1.5 |
| Calcium | 310 | <18 | 160 | 210 | 170 | <130 | 50 | 92 |
| Cadmium | <0.5 | <3.2 | <3.3 | <2.8 | <3.0 | <3.4 | <3.3 | <3.3 |
| Cobalt | <1.2 | <4.2 | <4.3 | <3.6 | <3.0 | <4.4 | <4.3 | <4.3 |
| Chromium | 2.1 | <5.1 | <5.3 | <4.4 | <4.8 | <6.6 | <5.3 | <5.3 |
| Copper | <2.5 | <11 | <12 | <9.7 | <10 | <12 | <12 | <12 |
| Iron | 250 | 102 | 78 | 260 | 120 | 110 | 74 | 1,100 |
| Magnesium | 84 | 19 | 73 | 92 | 72 | 57 | 11 | 63 |
| Manganese | 6.9 | 1.2 | 1.0 | 4.6 | 4.1 | 1.5 | 1.1 | 3.3 |
| Molybdenum | <2.5 | <7.6 | <7.8 | <6.6 | <7.0 | <8.0 | <7.8 | <7.8 |
| Sodium | 120 | <4,800 | <5,000 | <4,200 | <4,500 | <5,100 | <5,000 | <4,900 |
| Nickel | 89 | 110 | 92 | 100 | 92 | 120 | 100 | 100 |
| Lead | 7.9 | <53 | <55 | <46 | <49 | <56 | <55 | <54 |
| Tin | <4.9 | <1,100 | <1,100 | <920 | <990 | <1,100 | <1,100 | <1,100 |
| Titanium | <2.5 | <3.0 | <3.1 | 3.4 | 4.0 | <3.2 | <3.1 | <3.1 |
| Vanadium | 280 | 3,600 | 280 | 330 | 270 | <140 | 280 | 300 |
| Zinc | 10 | 9.3 | 5.1 | 8.2 | 7.1 | 5.1 | 5.9 | 4.8 |

TABLE 4-18. CHLORIDE AND SULFUR RESULTS FOR LIME PRODUCT, LIMESTONE FEED, AND BAGHOUSE DUST

| Run No. | Run condition | Chloride content, % | | | Sulfur content, %[a] | | Mass rate | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | Lime product | Limestone feed | Baghouse dust | Lime product | Baghouse dust | Lime[b] product, kg/hr | Limestone feed | | Baghouse dust,[c] kg/hr |
| | | | | | | | | kg/hr | lb/hr | |
| 1 | Baseline | 0.055 | 0.150 | 0.375 | 0.04 | 3.92 | 7,037[d] | 14,074 | 31,000 | 1,055 |
| 2 | Baseline | 0.105 | 0.175 | 0.725 | 0.05 | 2.35 | 7,037[d] | 14,074 | 31,000 | 1,055 |
| 3 | Baseline | 0.085 | 0.140 | 0.750 | 0.15 | 1.41 | 7,037[d] | 14,074 | 31,000 | 1,055 |
| 4 | Waste fuel | 0.080 | 0.088 | 1.750 | 0.09 | 2.96 | 7,080 | 14,160 | 31,200 | 1,062 |
| 5 | Waste fuel | 0.085 | 0.140 | 0.700 | 0.14 | 1.74 | 7,440 | 14,880 | 32,770 | 1,116 |
| 6[e] | Waste fuel | 0.085 | 0.133 | 1.363 | 0.10 | 1.34 | 8,285 | 16,570 | 36,500 | 1,243 |
| 7 | Waste fuel | 0.095[f]<br>0.070[g] | 0.150 | 0.800 | 0.10[f]<br>0.05[g] | 0.99 | 8,215[f]<br>8,580[g] | 16,430[f]<br>17,160[g] | 36,200[f]<br>37,800[g] | 1,232[f]<br>1,287[g] |
| 8 | Waste fuel | 0.070 | 0.075 | 1.225 | 0.04 | 0.85 | 8,650 | 17,300 | 38,130 | 1,298 |

[a] No sulfur analyses on limestone feed.

[b] Product rate assumed 50% of limestone feed rate.

[c] Baghouse dust rate assumed 15% of limestone rate.

[d] Assumed limestone feed, lime product and baghouse dust rates.

[e] Average of duplicate samples.

[f] Lime product sample taken prior to 1100 hours at low waste fuel feed rate.

[g] Lime product sample taken after 1100 hours at high waste fuel rate.

Chloride content was low in the lime product and limestone feed during both baseline and waste fuel conditions. Sulfur was not analyzed in the limestone feed. Sulfur content in the lime product was low compared to the baghouse dust levels (lime product average = 0.08%; baghouse dust average = 1.95%). The mass rate for the limestone feed is an average of hourly readings. The lime product rate was commonly 505 of the stone feed rate, and the baghouse dust rate was assumed to be 15% of the product rate, as described by Rockwell Lime personnel to MRC personnel during testing.

Trace metal results for the limestone feed, lime product, and baghouse dust are shown in Tables 4-19, 4-20, and 4-21, respectively. In all three of those samples, the metals of highest concentration include calcium, magnesium, sodium, iron, and aluminum.

Two lime product samples were taken on Day 7 during low and high waste fuel feed rates to evaluate possible change in the lime product. Table 4-18 indicates little or no change in chloride and lime concentrations and Table 4-20 also indicates no change in metals concentrations on day 7 from low waste fuel to high waste fuel feed rates.

## TABLE 4-19. TRACE METAL RESULTS FOR LIMESTONE FEED SAMPLES

| | Concentration, μg/g | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| Metal | Run No. 1 | Run No. 2 | Run No. 3 | Run No. 4 | Run No. 5 | Run No. 6[a] | Run No. 7 | Run No. 8 |
| Aluminum | 950 | 150 | 190 | 150 | 140 | 140 | 150 | 180 |
| Boron | <25 | <5.2 | <5.3 | <5.2 | <5.3 | <5.3 | <5.2 | <5.3 |
| Barium | 11 | <13 | <13 | <13 | <13 | <13 | <13 | <13 |
| Beryllium | <5.0 | 1.7 | 1.7 | 1.6 | 1.7 | 1.7 | 1.7 | 2.2 |
| Calcium | 210,000 | 220,000 | 220,000 | 210,000 | 220,000 | 220,000 | 220,000 | 220,000 |
| Cadmium | <5.0 | 1.7 | <1.7 | <1.7 | <1.7 | <1.7 | <1.7 | <1.7 |
| Cobalt | <13 | <2.2 | 2.9 | 2.3 | <2.2 | <2.6 | 2.2 | <2.2 |
| Chromium | <13 | 55 | 52 | 51 | 51 | 52 | 53 | 71 |
| Copper | <25 | 34 | 35 | 34 | 34 | 35 | 35 | 35 |
| Iron | 1,700 | 800 | 1,000 | 670 | 700 | 840 | 920 | 2,000 |
| Magnesium | 120,000 | 150,000 | 140,000 | 140,000 | 140,000 | 140,000 | 150,000 | 150,000 |
| Manganese | 110 | 100 | 100 | 93 | 100 | 110 | 110 | 110 |
| Molybdenum | <25.0 | <4.0 | <4.0 | <4.0 | <4.0 | <4.0 | <4.0 | <4.0 |
| Sodium | 250 | 3,500 | 3,400 | 3,200 | 3,000 | 3,500 | 3,200 | 2,500 |
| Nickel | <25.0 | 36 | 35 | 35 | 34 | 35 | 35 | 42 |
| Lead | <1.3 | <27 | <28 | <28 | <28 | <28 | <28 | <28 |
| Tin | <5.0 | <550 | <560 | <550 | <560 | <560 | <550 | <560 |
| Titanium | <25 | <1.6 | <1.6 | <1.6 | <1.6 | <1.6 | <1.6 | <1.6 |
| Vanadium | 10 | 23 | 22 | 19 | 20 | 22 | 22 | 35 |
| Zinc | <13 | <0.5 | 24 | 13 | 3.9 | 2.6 | 2.2 | 4.4 |

[a] Average of duplicate samples.

# TABLE 4-20. TRACE METAL RESULTS FOR LIME PRODUCT SAMPLES

| Metal | Concentration, µg/g | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | Run No. 1 | Run No. 2[a] | Run No. 3[a] | Run No. 4 | Run No. 5 | Run No. 6[b] | Run No. 7A[c] | Run No. 7B[d] | Run No. 8 |
| Aluminum | 70 | 380 | 300 | 760 | 260 | 550 | 216 | 350 | 380 |
| Boron | 25.0 | <5.3 | <5.3 | <5.2 | <5.3 | <5.3 | <5.3 | <5.3 | <5.2 |
| Barium | <5.0 | <13 | <13 | <13 | <13 | <13 | <13 | <13 | <13 |
| Beryllium | <5.0 | 2.1 | 2.0 | 2.0 | 2.0 | 2.0 | 2.3 | 2.4 | 2.4 |
| Calcium | 380,000 | 400,000 | 400,000 | 390,000 | 400,000 | 400,000 | 400,000 | 390,000 | 400,000 |
| Cadmium | <5.0 | <1.7 | <1.7 | <1.7 | <1.7 | <1.8 | <1.7 | <1.7 | <1.7 |
| Cobalt | <13 | 3.0 | 2.7 | 3.1 | 3.1 | 3.9 | <2.2 | 3.5 | <2.2 |
| Chromium | <13 | 70 | 68 | 68 | 67 | 67 | 72 | 81 | 76 |
| Copper | <25 | 49 | 48 | 47 | 49 | 49 | 44 | 47 | 47 |
| Iron | 1,700 | 1,500 | 1,400 | 1,500 | 1,300 | 1,600 | 1,300 | 1,600 | 1,900 |
| Magnesium | 220,000 | 267,500 | 272,500 | 260,000 | 270,000 | 260,000 | 280,000 | 270,000 | 270,000 |
| Manganese | 170 | 180 | 180 | 180 | 180 | 190 | 170 | 170 | 190 |
| Molybdenum | <25. | <4.0 | <4.0 | <4.0 | <4.0 | <4.0 | <4.0 | <4.0 | <4.0 |
| Sodium | 170 | 3,800 | 3,600 | 3,900 | 3,900 | 4,100 | <2,500 | <2,500 | <2,500 |
| Nickel | <25 | 47 | 45 | 43 | 44 | 45 | 47 | 50 | 49 |
| Lead | <1.3 | <28 | <28 | 82 | <28 | <28 | <28 | <28 | <28 |
| Tin | <5.0 | <560 | <560 | <550 | <560 | <560 | <560 | <560 | <550 |
| Titanium | 25 | <1.6 | <1.6 | <1.6 | <1.6 | <45.5 | <1.6 | 28.7 | <1.6 |
| Vanadium | <10 | 33 | 31 | 30 | 31 | 31 | 36 | 39 | 40 |
| Zinc | 9.5 | 4.0 | 2.0 | 4.0 | 6.9 | 11.2 | 1.5 | 8.0 | 6.5 |

[a] Average of split analyses.

[b] Average of three analyses.

[c] Sample taken before 1100 hours.

[d] Sample taken after 11000 hours.

## TABLE 4-21. TRACE METAL RESULTS FOR BAGHOUSE DUST SAMPLES

| Metal | Concentration, μg/g | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | Run No. 1 | Run No. 2 | Run No. 3 | Run No. 4 | Run No. 5 | Run No. 6[a] | Run No. 7 | Run No. 8 |
| Aluminum | 1,000 | 830 | 770 | 950 | 840 | 980 | 580 | 610 |
| Boron | <25 | <5.3 | <5.3 | 11 | 7.3 | 13 | <5.3 | 7.4 |
| Barium | 8.5 | <13 | <13 | 15 | <13 | 28 | <13 | <13 |
| Beryllium | <5.0 | 3.6 | 2.6 | 3.9 | 2.7 | 3.0 | 2.8 | 2.8 |
| Calcium | 230,000 | 260,000 | 270,000 | 260,000 | 270,000 | 290,000 | 290,000 | 290,000 |
| Cadmium | <5.0 | <1.7 | <1.7 | 1.9 | <1.7 | <1.8 | <1.7 | 1.7 |
| Cobalt | <13 | <2.2 | <2.2 | 8.3 | 7.6 | 15 | 6.1 | 11 |
| Chromium | <13 | 71 | 67 | 160 | 140 | 220 | 120 | 150 |
| Copper | <25 | 39 | 35 | 46 | 44 | 55 | 45 | 47 |
| Iron | 8,500 | 1,700 | 1,600 | 2,500 | 2,100 | 2,700 | 1,700 | 2,000 |
| Magnesium | 130,000 | 160,000 | 160,000 | 170,000 | 170,000 | 180,000 | 190,000 | 200,000 |
| Manganese | 130 | 130 | 130 | 130 | 130 | 140 | 120 | 120 |
| Molybdenum | <25. | <4.0 | <4.0 | <4.0 | <4.0 | <4.0 | <4.0 | <4.0 |
| Sodium | 700 | 2,900 | <2,500 | 3,800 | 3,100 | 4,100 | 3,200 | 3,700 |
| Nickel | 43 | 160 | 74 | 170 | 87 | 100 | 97 | 91 |
| Lead | 1.7 | <28 | <28 | 200 | 140 | 370 | 160 | 270 |
| Tin | <5.0 | <560 | <560 | <560 | <560 | <560 | <560 | <560 |
| Titanium | 26 | <1.6 | <1.6 | 630 | 640 | 1,500 | 470 | 820 |
| Vanadium | 120 | 350 | 130 | 400 | 150 | 190 | 180 | 160 |
| Zinc | 11 | 9.7 | 5.3 | 54 | 38 | 84 | 46 | 89 |

[a] Average of duplicate samples.

# SECTION 5

# ANALYSIS OF RESULTS

## 5.1 DESTRUCTION AND REMOVAL EFFICIENCIES

The complex combustion chemistry for organic materials becomes perplexing when a broad range of organic compounds present in a liquid waste are burned. On a weight basis, most of the organic carbon in the waste is oxidized to $CO_2$ in the combustion process, but trace amounts of organic chemicals survive the oxidation process and are only partially reacted. Accordingly, the test burn investigated the amount of destruction of the organic compounds in the hazardous waste.

Destruction and removal efficiency (DRE) for an incineration/air pollution control system is defined by the following equation:

$$DRE = \frac{W_{in} - W_{out}}{W_{in}} (100) \qquad (5\text{-}1)$$

where DRE = destruction and removal efficiency, %

$W_{in}$ = mass feed rate of principal organic hazardous constituent(s) (POHCs) fed to the incinerator

$W_{out}$ = mass emission rate of principal organic hazardous constituent(s) (POHCs) to the atmosphere (as measured in stack prior to discharge).

DRE calculations are based on combined efficiencies of the destruction of the POHC in the incinerator, or the lime kiln, and the removal of the POHC from the gas stream in the air pollution control system. The presence of POHCs in solid discharges from the air pollution control devices is not accounted for in the DRE calculation as currently defined by the EPA. RCRA Part 264, Subpart O regulations for hazardous waste incinerators require a DRE of 99.99% for all principal organic hazardous constituents of a waste during trial burns unless it can be demonstrated that a higher or lower DRE is more appropriate based on human health criteria. However, lime kilns are currently exempted from trial burn testing based on federal regulations. Specification of the POHCs in a waste is subject to best engineering judgment, considering the toxicity, thermal stability, and quantity of each organic waste constituent. DRE requirements in the Subpart O regulations do not apply to metals or other noncombustible materials.

The POHCs chosen in this test program were six compounds known to be present in the current fuel mixture:

- Methylene chloride ($MeCl_2$)
- Methyl ethyl ketone (MEK)
- 1,1,1-Trichloroethane ($CH_3CCl_3$)
- Trichloroethylene (TCE)
- Tetrachloroethylene (Perc)
- Toluene

Toluene, MEK, Perc, and TCE were present in high concentration for organic compounds (see Table 4-13). Spikes of Perc and TCE were added to the waste fuel prior to the test burn to obtain the higher concentrations. Perc, TCE, methylene chloride, and 1,1,1-trichloroethane were selected because the chlorinated hydrocarbons are, in general, difficult to thermally destroy. All six of the compounds, except methylene chloride, were found in the top twenty constituents of the waste fuel. All six compounds are listed hazardous compounds in RCRA Part 261, Appendix VIII.

These six POHCs were sampled in the exhaust gas by the volatile organic sampling train (VOST) and analyzed by gas chromatography/mass spectrometry (GC/MS). Results of the analysis for exhaust gas concentration of POHCs ($W_{out}$) were presented in Table 4-11.

Grab samples of waste fuel feed were composited for daily samples and analyzed by GC/MS. Results of the mass feed rate for each POHC ($W_{in}$) were presented in Table 4-14.

The DREs calculated using Equation 5-1 for each test run for POHCs are shown in Table 5-1. These results are calculted using the $W_{out}$ values listed in Table 4-11, which assume that the tube blanks equal zero and $W_{in}$ values listed in Table 4-14. If tube blanks were included in the calculation, $W_{out}$ values would be less and, thus DREs would be higher. Therefore, the DREs presented in Table 5-1 are based on a worst-case assumption.

DREs were determined only for Run Nos. 4 through 8 when waste fuel was being burned. During Run Nos. 1 through 3, $W_{in}$ equals zero; thus, a DRE can not be determined. A comparison of baseline vs. waste fuel conditions for POHC emissions and other pollutants are presented in Section 5.5.

In general, DREs ranged from 99.60% to >99.999% for all compounds and averaged 99.9989%. Only four runs had DREs less than 99.99%; three of these were for methylene chloride, the fourth for 1,1,1,-trichloroethane.

DREs for methylene chloride ranged from 99.60% to <99.999% and averaged 99.983% ± 0.15% (95% confidence limits). Three runs were less than 99.99% (Run Nos. 5A, 5F, and 4E). In Run No. 5A, the tube mass collected was 1,066 ng compared to a maximum of 55 ng for all of the other runs. This high mass was most likely caused by lab contamination. Run No. 5A is not included in the averages, due to the lab contamination, thus the Day 5 average is 99.995% and the overall methylene chloride DRE average is 99.997%. Contamination of any individual sample by methylene chloride was likely, as methylene chloride is commonly used as an extraction solvent in the MRC Dayton Laboratory. Because of potential methylene chloride contamination, these results should be viewed with caution.

## TABLE 5-1. DESTRUCTION AND REMOVAL EFFICIENCIES (DREs) FOR POHCS

| Run number | DRE, %[a] | | | | | |
|---|---|---|---|---|---|---|
| | $MeCl_2$ | MEK | $CH_3$ $CCl_3$ | TCE | Perc | Toluene |
| 4A | 99.9971 | 99.9996 | 99.9980 | 99.9998 | 99.9998 | 99.9997 |
| 4B | 99.9978 | 99.9991 | 99.9963 | 99.9998 | 99.9998 | 99.9997 |
| 4C | 99.9996 | 99.9994 | 99.9981 | 99.9997 | 99.9998 | 99.9998 |
| 4D | 99.9932 | 99.9987 | 99.9929 | 99.9999 | 99.9999 | 99.9997 |
| 4E | 99.9847 | 99.9995 | 99.9894 | 99.9998 | 99.9998 | 99.9997 |
| 4F | 99.9959 | 99.9999 | 99.9985 | 99.9999 | 99.9999 | 99.9999 |
| Day 4 average | 99.9947 | 99.9994 | 99.9955 | 99.9998 | 99.9998 | 99.9998 |
| 5A | 99.5979[b] | 99.9998 | 99.9940 | 99.9997 | 99.9999 | 99.9999 |
| 5B | 99.9995 | 99.9999 | 99.9997 | 99.9999 | 99.9999 | 99.9999 |
| 5C | 99.9984 | 99.9993 | 99.9994 | 99.9999 | 99.9999 | 99.9998 |
| 5D | 99.9995 | 99.9997 | 99.9994 | 99.9998 | 99.9999 | 99.9998 |
| 5E | 99.9941 | 99.9995 | 99.9980 | 99.9997 | 99.9998 | 99.9995 |
| 5F | 99.9828 | 99.9996 | 99.9989 | 99.9998 | 99.9999 | 99.9997 |
| Day 5 average | 99.9949 | 99.9996 | 99.9982 | 99.9997 | 99.9999 | 99.9998 |
| 6A | 99.9998 | 99.9997 | 99.9997 | 99.9999 | 99.9999 | 99.9999 |
| 6B | 99.9999 | 99.9995 | 99.9997 | 99.9999 | 99.9999 | 99.9999 |
| 6C | 99.9981 | 99.9999 | 99.9913 | 99.9997 | 99.9999 | 99.9999 |
| 6D | 99.9998 | 99.9998 | 99.9994 | 99.9999 | 99.9999 | 99.9998 |
| 6E | 99.9991 | 99.9999 | 99.9978 | 99.9999 | 99.9998 | 99.9997 |
| 6F | 99.9994 | 99.9999 | 99.9973 | 99.9997 | 99.9998 | 99.9997 |
| Day 6 average | 99.9994 | 99.9997 | 99.9975 | 99.9998 | 99.9999 | 99.9998 |
| 7A | 99.9993 | 99.9995 | 99.9980 | 99.9999 | 99.9999 | 99.9996 |
| 7B | 99.9983 | 99.9987 | 99.9975 | 99.9998 | 99.9989 | 99.9995 |
| 7C | 99.9991 | 99.9979 | 99.9905 | 99.9998 | 99.9995 | 99.9994 |
| 7D | 99.9991 | 99.9996 | 99.9993 | 99.9999 | 99.9999 | 99.9996 |
| 7E | 99.9992 | 99.9999 | 99.9944 | 99.9997 | 99.9999 | 99.9994 |
| 7F | 99.9962 | 99.9998 | 99.9977 | 99.9999 | 99.9998 | 99.9997 |
| Day 7 average | 99.9985 | 99.9992 | 99.9962 | 99.9999 | 99.9997 | 99.9995 |
| 8A | 99.9995 | 99.9999 | 99.9986 | 99.9998 | 99.9998 | 99.9996 |
| 8B | 99.9991 | 99.9996 | 99.9976 | 99.9999 | 99.9997 | 99.9998 |
| 8C | 99.9997 | 99.9999 | 99.9973 | 99.9998 | 99.9996 | 99.9997 |
| 8D | 99.9997 | 99.9992 | 99.9943 | 99.9996 | 99.9997 | 99.9997 |
| Day 8 average | 99.9995 | 99.9997 | 99.9969 | 99.9998 | 99.9997 | 99.9997 |
| Overall average | 99.9974 | 99.9995 | 99.9969 | 99.9998 | 99.9998 | 99.9997 |

[a] % DRE = $(W_{in} - W_{out})/W_{in} \times 100$; assume tube blanks = 0 ng.

[b] Contaminated sample, not included in average for Day 5 or overall testing.

Methyl ethyl ketone had an average DRE of 99.999% ± 0.0002% (95% confidence limit) and ranged from 99.998% to greater than 99.999%. These high destruction efficiencies were consistent throughout the waste fuel test runs.

DREs for 1,1,1-trichloroethane ranged from 99.98 % to 99.99 % and averaged 99.997% ± 0.004% (95% confidence limits). Only Run No. 4E had a DRE less than 99.99%. For many sample runs, the sampling method and duration used for measuring 1,1,1-trichloroethane in the stack gas was not able to detect the 1,1,1-trichloroethane since it was at such extremely low concentrations. For example, the concentration of 1,1,1-trichloroethane in the waste fuel was low (average concentration of 0.24% by weight) such that at high destruction effeciencies the mass of 1,1,1-trichloroethane collected in the stack was often less than the sample tube blanks. To obtain a greater mass of 1,1,1-trichloroethane on the tubes, either a higher waste fuel concentration or a longer sampling duration may have been helpful. However, maximum sample duration/volume was limited based on volumetric gas capacity (Vg) [5].

DREs for trichloroethylene (TCE) were greater than 99.99 % for all runs. TCE was spiked to the waste fuel prior to the waste fuel testing to increase its concentration and allow easier detection of TCE in the stack gas. Spiking of TCE to concentrations greater than approximately 1.3% by volume was not possible due to permit requirements which specified a maximum of 3.0% by volume for chlorine. As in 1,1,1-trichloroethane, the mass of TCE collected from several runs was less than the tube blanks. This indicates that very high DREs (>99.999%) for TCE were obtained.

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[5] Values for Vg and sample volumes for each POHC were estimated prior to the test; refer to Reference [3] and Section 3.3.1.

DREs for tetrachloroethylene (Perc) also were greater than 99.999% for all runs. Like TCE, Perc was spiked to the waste fuel to the maximum allowable concentration described in the test burn permit prior to the test.

Toluene was the POHC of highest concentration in the waste fuel (average 11.6% by weight). DREs for toluene were above 99.999% and were consistent for all test runs.

Based on Tables 4-9 (tube mass values) and 4-10 (tube blank mass values), many tube masses were less than the tube blank mass. The cause for the tube values being less than blank tube values was a combination of the low waste fuel concentration of several of the POHCs and high DREs resulting in emission rates lower than originally anticipated. In addition, blank values were slightly higher than expected. To obtain tube masses greater than blank values either the POHC waste fuel concentration should have been higher or the DRE lower, neither of which were possible or practical to effect during the test, or additional sample volume should have been collected. However, maximum sample volume was limited based on tube/POHC volumetric capacity and to avoid breakthroughs caution was exercised when sample volumes and rates were selected.

## 5.2 CHLORINE AND SULFUR MATERIAL BALANCES

The combustion of chlorinated hazardous wastes as fuel in lime kilns results in the generation of hydrogen chloride (HCl) in the kiln. Hydrogen chloride can be absorbed by the lime product or kiln lining, removed in the baghouse, or emitted with the particulate. Nonabsorbed HCl is emitted from the stack as is the unburned chlorinated hydrocarbons. The chlorine material balance is presented to demonstrate the fate of chlorine in the kiln process.

A schematic diagram for the lime kiln is shown in Figure 5-1. The material balance for chlorine (as well as for sulfur and metals) used the following process streams:

| Inputs | Outputs |
|---|---|
| Coke | Lime product |
| Waste fuel | Baghouse dust |
| Limestone feed | Stack gas |

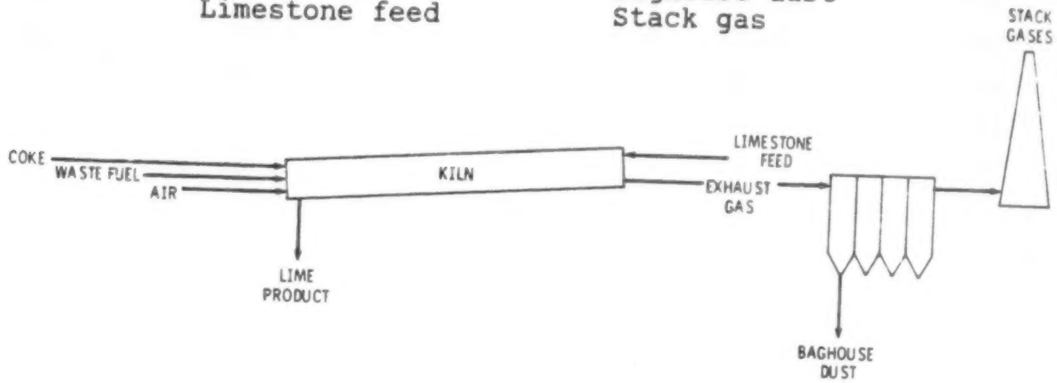

Figure 5-1. Schematic diagram of lime kiln process for material balance.

Table 5-2 shows the results of the chlorine material balance. Material balance closure (total out ÷ total in) ranged from 36% to 188% and averaged 88% (100% closure is ideal). Possible explanations for closures not being ideal are: (1) chlorine absorbed by the kiln walls, (2) chlorine in the particulate, (3) random error associated with chlorine sampling and analytical results, and (4) errors in estimating the flow rates of the process streams.

In general, most of the chlorine is introduced into the kiln in the limestone feed (typically greater than 90% of total input chlorine). Most of the chlorine exiting the kiln was contained in the lime product (23% to 49%) and in the baghouse dust (50% to 76%). Typically, the chlorine in the stack gas was a very low percentage of the total chlorine emitted from the kiln (less than 1.6%).

TABLE 5-2. CHLORINE MATERIAL BALANCE

| Run number | Materials to kiln, kg/hr | | | | Materials from kiln, kg/hr | | | | Material balance closure, % |
|---|---|---|---|---|---|---|---|---|---|
| | Coke | Waste fuel | Limestone feed | Total in | Lime product | Baghouse dust | Stack gas | Total out | |
| 1 | 0.567 | 0 | 21.24 | 21.81 | 3.89 | 3.98 | 0.054 | 7.92 | 36 |
| 2 | 0.552 | 0 | 24.78 | 25.33 | 7.43 | 7.70 | 0.091 | 15.22 | 60 |
| 3 | 1.03 | 0 | 19.82 | 20.85 | 6.02 | 7.96 | 0.169 | 14.15 | 68 |
| 4 | 0.354 | 0.136 | 12.46 | 12.95 | 5.66 | 18.59 | 0.115 | 24.37 | 188 |
| 5 | 0.381 | 0.220 | 20.83 | 21.43 | 6.32 | 7.81 | 0.193 | 14.32 | 67 |
| 6 | 0.640 | 0.423 | 22.04 | 23.10 | 7.04 | 16.94 | 0.231 | 24.21 | 105 |
| 7 | 0.576[a] | 0.154 | 24.65 | 25.38 | 7.80 | 9.86 | 0.149 | 17.81 | 70 |
| | 0.478[b] | 0.573 | 25.74 | 26.79 | 6.01 | 10.30 | 0.261 | 16.57 | 62 |
| 8 | 2.60 | 0.644 | 12.98 | 16.22 | 6.01 | 15.90 | 0.220 | 22.13 | 136 |

[a] Prior to 1100 hours.

[b] After 1100 hours.

Although not part of the program goals, all process streams, except the limestone feed, were analyzed for sulfur. Using the sulfur analytical results and process mass rates, a material balance for sulfur was completed, and results are shown in Table 5-3. For each test a material balance closure was calculated assuming both a zero sulfur value and a 0.1% sulfur value for the limestone feed. In the first case (S = 0% in limestone), balance closures ranged from 41% to 101% and averaged 76%. In the second case (S = 0.1% in limestone), the balance closure ranged from 51% to 123%, and averaged 98%. In either case, the majority of the sulfur is introduced to the kiln in the coke feed (80% to 100% total sulfur), and very little sulfur is introduced in the waste fuel (<0.5%).

TABLE 5-3. SULFUR MATERIAL BALANCE

| | Materials to kiln, kg/hr | | | | Materials from kiln, kg/hr | | | | Material balance closure, % |
|---|---|---|---|---|---|---|---|---|---|
| Run number | Coke | Waste fuel | Limestone feed | Total in | Lime product | Baghouse dust | Stack gas | Total out | |
| 1 | 56.96 | 0 | NA[a] | 56.96 | 2.82 | 41.36 | 94.43 | 138.6 | 41 |
| | | | ∼14.07[b] | 71.03 | | | | | 51 |
| 2 | 57.39 | 0 | NA[a] | 57.39 | 3.52 | 24.79 | 45.00 | 73.31 | 78 |
| | | | ∼14.07[b] | 71.46 | | | | | 97 |
| 3 | 62.19 | 0 | NA[a] | 62.19 | 10.56 | 14.88 | 36.34 | 61.78 | 101 |
| | | | ∼14.07[b] | 76.26 | | | | | 123 |
| 4 | 54.75 | 0.18 | NA[a] | 54.93 | 6.37 | 31.44 | 35.11 | 73.72 | 74 |
| | | | ∼14.16[b] | 69.09 | | | | | 94 |
| 5 | 54.75 | 0.23 | NA[a] | 54.98 | 10.42 | 19.42 | 37.72 | 67.56 | 81 |
| | | | ∼14.88[b] | 69.86 | | | | | 103 |
| 6 | 55.89 | 0.31 | NA[a] | 56.20 | 8.29 | 16.66 | 43.34 | 68.29 | 82 |
| | | | ∼16.57[b] | 72.77 | | | | | 107 |
| 7A | 61.55 | 0.10 | NA[a] | 61.65 | 8.22 | 12.20 | 66.36 | 86.78 | 71 |
| | | | ∼16.43[b] | 78.08 | | | | | 90 |
| 7B | 51.12 | 0.39 | NA[a] | 51.51 | 4.29 | 12.74 | 40.83 | 57.86 | 89 |
| | | | ∼17.16[b] | 68.67 | | | | | 119 |
| 8 | 45.26 | 0.70 | NA[a] | 45.96 | 3.46 | 11.03 | 52.03 | 66.52 | 69 |
| | | | ∼17.30[b] | 63.26 | | | | | 95 |

[a] NA = Not available.

[b] Assumes 0.1% sulfur in limestone feed.

The sulfur exits the kiln in the following streams and approximate proportions: stack gas (50% to 80%), baghouse dust (15% to 35%), and lime product (2% to 15%).

## 5.3 MATERIAL BALANCE FOR METALS

The metals fed to the kiln in the coke, waste fuel, and limestone feed (refer to Figure 5-1) can be absorbed by the lime product or kiln lining, removed by the baghouse, or emitted as part of the stack gas particulates. Nonabsorbed metals will be emitted by the stack. A material balance for metals is presented to demonstrate the fate of the metals in the kiln process.

Tables 5-4 through 5-11 show the distribution of metals for each run. Because the concentrations of several metals in the various input and output streams were below the limit of quantification (LOQ) for the ICAP analysis, mass emission rates are often reported as "less than" (<) values, and material balances were not calculated for several metals.

Metals for which material balances were calculated include aluminum, beryllium, calcium, cobalt, chromium, copper, iron, magnesium, manganese, nickel, lead, tin, vanadium, and zinc. The material balance closure was calculated by dividing the total mass out by total mass in. The range of material balance closures for all of the metals and all runs was between <6% to 291%, and the average was 87% ± 77% (95% confidence limits). Material balance closures close to 100% occurred for calcium, magnesium, and manganese. The remaining metals displayed considerable variability in material balance results.

Causes for the material balance results not being 100% include the nonmeasurement of metals emitted as nonparticulates from the stack, metals absorbed by the kiln refractory, errors in the measurement of flow rates for input and output materials, and random error associated with metal sampling and analytical results.

In general, the metals are introduced to the kiln primarily in the limestone feed and according to the following proportions: limestone feed >83%, coke 5%-20%, waste fuel <5%. The following metals are exceptions: nickel and lead where limestone feed has 75%-85%, coke has 13%-25%, and waste fuel has <2%; vanadium where limestone feed has 37%-84%, coke has 40%-70%, and waste fuel has <2%; and zinc where limestone feed has 30%-80%, coke has 20%-70%, and waste fuel has 0%-10%. The metals generally exit the kiln system in the lime product (70% to 90% of total emissions in lime

## TABLE 5-4. MATERIAL BALANCE FOR METALS, RUN NO. 1

|  | Materials to kiln, g/hr |  |  |  | Materials from kiln, g/hr |  |  |  |  |
|---|---|---|---|---|---|---|---|---|---|
| Metal | Coke | Waste fuel | Limestone feed | Total in | Lime product | Baghouse dust | Stack gas particulates[a] | Total out | Material balance closure[b], % |
| Aluminum | 79 | NA[c] | 13,000 | 13,000 | 490 | 1,100 | <4.1 | 1,600 | 12 |
| Boron | <3.6 |  | <350 | <360 | 180 | <26 | <0.3 | 210 |  |
| Barium | 3.1 |  | 150 | 150 | <35 | 9.0 | <0.7 | <45 |  |
| Beryllium | <0.7 |  | <70 | <71 | <35 | <5.3 | 0 | <40 |  |
| Calcium | 450 |  | 2,900,000 | 2,900,000 | 2,700,000 | 240,000 | 230 | 2,900,000 | 100 |
| Cadmium | <0.7 |  | <70 | <71 | <35 | <5.3 | <0.1 | <40 |  |
| Cobalt | <1.7 |  | <180 | <180 | <88 | <13 | <0.2 | <100 |  |
| Chromium | 3.1 |  | <180 | <180 | <88 | <13 | 3.1 | <100 |  |
| Copper | <3.6 |  | <350 | <350 | <180 | <26 | <0.7 | <210 |  |
| Iron | 360 |  | 24,000 | 24,000 | 12,000 | 9,000 | 8.8 | 21,000 | 88 |
| Magnesium | 120 |  | 1,700,000 | 1,700,000 | 1,500,000 | 130,000 | 160 | 1,600,000 | 94 |
| Mangenese | 10 |  | 1,600 | 1,600 | 1,200 | 140 | 0.3 | 1,300 | 81 |
| Molybdenum | <3.6 |  | <350 | <350 | <180 | <26 | <0.2 | <210 |  |
| Sodium | 180 |  | 3,500 | 3,700 | 1,200 | 740 | <5.5 | 1,900 | 51 |
| Nickel | 130 |  | <350 | <480 | <180 | 45 | 0.9 | 230 |  |
| Lead | 12 |  | <18 | <30 | <9.1 | <1.8 | <1.4 | <12 |  |
| Tin | <7.1 |  | <70 | <77 | <35 | <5.3 | <17 | <57 |  |
| Titanium | <3.6 |  | <350 | <350 | 180 | 27 | 0.2 | 210 |  |
| Vanadium | 410 |  | 140 | 550 | <70 | 130 | 1.3 | 200 | 36 |
| Zinc | 15 |  | <180 | <200 | 67 | 12 | 0.1 | 79 |  |

[a] Average of two samples.

[b] Blanks indicate value not calculated.

[c] No waste fuel fired during this run.

## TABLE 5-5. MATERIAL BALANCE FOR METALS, RUN NO. 2

| | Materials to kiln, g/hr | | | | Materials from kiln, g/hr | | | | |
|---|---|---|---|---|---|---|---|---|---|
| Metal | Coke | Waste fuel | Limestone feed | Total in | Lime product | Baghouse dust | Stack gas particulates | Total out | Material balance closure[a], % |
| Aluminum | 140 | NA[b] | 2,100 | 2,200 | 2,700 | 870 | 6.4 | 3,600 | 164 |
| Boron | <15 | | <73 | 88 | <37 | <5.6 | <0.6 | <43 | |
| Barium | <36 | | <180 | <220 | <91 | <14 | <0.7 | <110 | |
| Beryllium | 2.9 | | 24 | 27 | 15 | 3.8 | 0 | 19 | 70 |
| Calcium | <27 | | 3,100,000 | 3,100,000 | 2,800,000 | 270,000 | 220 | 3,100,000 | 100 |
| Cadmium | <4.7 | | <24 | <29 | <12 | <1.8 | <0.1 | <14 | |
| Cobalt | <6.1 | | <31 | <37 | 21 | <2.3 | 0.2 | 24 | |
| Chromium | <7.4 | | 780 | 790 | 490 | 75 | 1.9 | 570 | 72 |
| Copper | <16 | | 480 | 500 | 340 | 41 | 0.6 | 380 | 76 |
| Iron | 150 | | 11,000 | 11,000 | 11,000 | 1,800 | 5.2 | 13,000 | 118 |
| Magnesium | 28 | | 2,000,000 | 2,000,000 | 1,900,000 | 170,000 | 170 | 2,100,000 | 105 |
| Mangenese | 1.7 | | 1,400 | 1,400 | 1,300 | 140 | 0.3 | 1,400 | 100 |
| Molybdenum | 11 | | <56 | <67 | <28 | <4.2 | <0.2 | <32 | |
| Sodium | <7,000 | | 49,000 | 56,000 | 27,000 | 3,100 | <240 | 30,000 | 54 |
| Nickel | 160 | | 500 | 660 | 330 | 160 | 1.0 | 400 | 74 |
| Lead | <77 | | <380 | <460 | <200 | <29 | <1.5 | <230 | |
| Tin | <1,500 | | 7,800 | <9,300 | <3,900 | 590 | <9.9 | <4,500 | |
| Titanium | <4.4 | | <23 | 27 | <11 | <1.7 | 0.3 | <13 | |
| Vanadium | 520 | | 320 | 840 | 230 | 360 | 1.8 | 590 | 70 |
| Zinc | 14 | | <7.0 | 21 | 28 | 10 | 1.0 | 39 | 186 |

[a] Blanks indicate value not calculated.

[b] No waste fuel fired during this run.

## TABLE 5-6. MATERIAL BALANCE FOR METALS, RUN NO. 3

| | Materials to kiln, g/hr | | | | Materials from kiln, g/hr | | | | Material balance closure[a], % |
|---|---|---|---|---|---|---|---|---|---|
| Metal | Coke | Waste fuel | Limestone feed | Total in | Lime product | Baghouse dust | Stack gas particulates | Total out | |
| Aluminum | <140 | NA[b] | 2,600 | 2,700 | 2,800 | 810 | NA[c] | 3,600 | 133 |
| Boron | <15 | | <75 | <90 | <37 | <5.6 | | <43 | |
| Barium | <37 | | <180 | <220 | <91 | <14 | | <110 | |
| Beryllium | 2.2 | | 24 | 26 | 14 | 2.7 | | 17 | 65 |
| Calcium | 230 | | 3,000,000 | 3,000,000 | 2,900,000 | 280,000 | | 3,200,000 | 107 |
| Cadmium | <4.8 | | <24 | <29 | <12 | <1.8 | | <14 | |
| Cobalt | <6.3 | | 41 | 47 | 19 | <2.3 | | 21 | 45 |
| Chromium | <7.7 | | 730 | 740 | 480 | 71 | | 550 | 74 |
| Copper | <17 | | 490 | 510 | 340 | 37 | | 380 | 75 |
| Iron | 110 | | 14,000 | 14,000 | 9,800 | 1,700 | | 12,000 | 86 |
| Magnesium | 110 | | 2,000,000 | 2,000,000 | 1,900,000 | 160,000 | | 2,100,000 | 105 |
| Mangenese | 1.5 | | 1,400 | 1,400 | 1,300 | 140 | | 1,400 | 100 |
| Molybdenum | <11 | | <56 | <67 | <28 | <4.2 | | <32 | |
| Sodium | <7,200 | | 48,000 | 55,000 | 26,000 | <2,700 | | 29,000 | 53 |
| Nickel | 160 | | 500 | 660 | 310 | 78 | | 390 | 59 |
| Lead | <80 | | <390 | <470 | <2,000 | <29 | | <2,000 | |
| Tin | <1,600 | | <7,900 | <9,500 | <3,900 | <590 | | <4,500 | |
| Titanium | <4.5 | | <23 | <28 | <11 | <1.7 | | <13 | |
| Vanadium | 410 | | 310 | 720 | 220 | 130 | | 350 | 49 |
| Zinc | 7.4 | | 340 | 350 | 14 | 5.6 | | 20 | 6 |

[a] Blanks indicate value not calculated.

[b] No waste fuel fired during this run.

[c] No metals analysis made on Run No. 3.

## TABLE 5-7. MATERIAL BALANCE FOR METALS, RUN NO. 4

| | Materials to kiln, g/hr | | | | Materials from kiln, g/hr | | | | |
|---|---|---|---|---|---|---|---|---|---|
| Metal | Coke | Waste fuel | Limestone feed | Total in | Lime product | Baghouse dust | Stack gas particulates[a] | Total out | Material balance closure[b], % |
| Aluminum | <110 | <7.3 | 2,100 | 2,200 | 5,400 | 1,000 | 2.1 | 6,400 | 291 |
| Boron | <12 | 2.8 | <74 | <89 | <37 | 11 | <0.5 | <49 | |
| Barium | <29 | <1.9 | <180 | <210 | <91 | 16 | <0.7 | <110 | |
| Beryllium | 2.5 | 0 | 23 | 25 | 14 | 4.1 | 0 | 18 | 72 |
| Calcium | 280 | <1.4 | 3,000,000 | 3,000,000 | 2,700,000 | 280,000 | 120 | 3,000,000 | 100 |
| Cadmium | <3.8 | <0.2 | <24 | <28 | <12 | 2.0 | <0.1 | <14 | |
| Cobalt | <4.9 | 3.6 | 33 | <42 | 22 | 8.8 | 0.2 | 31 | 74 |
| Chromium | <6.0 | 1.6 | 720 | 730 | 480 | 170 | 1.7 | 650 | 89 |
| Copper | <13 | <0.9 | 480 | 490 | 330 | 49 | 0.6 | 380 | 78 |
| Iron | 350 | 4.3 | 9,500 | 9,000 | 10,000 | 2,700 | 7.5 | 13,000 | 131 |
| Magnesium | 130 | 0 | 2,000,000 | 2,000,000 | 1,800,000 | 180,000 | 88 | 2,000,000 | 100 |
| Mangenese | 6.3 | <0.3 | 1,300 | 1,300 | 1,200 | 140 | 0.2 | 1,300 | 100 |
| Molybdenum | <9.0 | <0.6 | <57 | <67 | <28 | 4.3 | <0.2 | <33 | |
| Sodium | <5,700 | <370 | 45,000 | 51,000 | 27,000 | 4,000 | <200 | 31,000 | 61 |
| Nickel | 140 | <1.0 | 490 | 630 | 300 | 180 | 1.0 | 480 | 76 |
| Lead | <63 | 19 | <390 | <470 | 580 | 210 | <1.5 | 790 | |
| Tin | <1,300 | <81 | <7,800 | <9,200 | <3,900 | <590 | <7.6 | <4,500 | |
| Titanium | 4.6 | 0.4 | <23 | <28 | <11 | 670 | 1.0 | 690 | |
| Vanadium | 450 | <0.5 | 310 | 760 | 210 | 420 | 1.1 | 630 | 83 |
| Zinc | 11 | 36 | 340 | 390 | 28 | 57 | 1.1 | 86 | 22 |

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[a] Average of two samples.

[b] Blanks indicate value not calculated.

[c] No waste fuel fired during this run.

TABLE 5-8. MATERIAL BALANCE FOR METALS, RUN NO. 5

| | Materials to kiln, g/hr | | | | Materials from kiln, g/hr | | | | |
|---|---|---|---|---|---|---|---|---|---|
| Metal | Coke | Waste fuel | Limestone feed | Total in | Lime product | Baghouse dust | Stack gas particulates[a] | Total out | Material balance closure[a], % |
| Aluminum | <120 | <14 | 2,100 | 2,200 | 2,000 | 930 | 5.1 | 2,900 | 132 |
| Boron | <13 | 10 | <79 | <100 | <39 | 8.1 | <0.5 | <48 | |
| Barium | <31 | <3.6 | <190 | <220 | <96 | <14 | <0.7 | <110 | |
| Beryllium | 1.9 | <0.1 | 25 | 27 | 15 | 3.0 | 0 | 18 | 67 |
| Calcium | 220 | <2.6 | 3,200,000 | 3,200,000 | 3,000,000 | 300,000 | 140 | 3,300,000 | 103 |
| Cadmium | <4.1 | <0.5 | <25 | <30 | <13 | <1.9 | <0.1 | <15 | |
| Cobalt | <5.3 | 6.4 | <32 | <45 | 23 | 8.5 | <0.2 | 32 | |
| Chromium | <6.5 | 3.2 | 760 | 770 | 500 | 150 | 2.1 | 650 | 84 |
| Copper | <14 | <1.6 | 510 | 530 | 360 | 50 | 0.5 | 410 | 77 |
| Iron | 170 | 9.3 | 10,000 | 10,000 | 9,400 | 2,300 | 5.7 | 12,000 | 120 |
| Magnesium | 98 | <0.1 | 2,100,000 | 2,100,000 | 2,000,000 | 190,000 | 96 | 2,200,000 | 105 |
| Mangenese | 5.6 | 0.6 | 1,500 | 1,500 | 1,300 | 150 | 0.3 | 1,500 | 100 |
| Molybdenum | <9.5 | <1.1 | <60 | <70 | <30 | <4.5 | <0.2 | <35 | |
| Sodium | <6,000 | <700 | 45,000 | 52,000 | 29,000 | 3,500 | <220 | 33,000 | 64 |
| Nickel | <130 | <2.0 | 500 | 630 | 330 | 97 | 0.9 | 430 | 68 |
| Lead | <67 | 36 | <420 | <520 | <210 | 160 | <1.8 | 370 | |
| Tin | <1,300 | <150 | <8,300 | <9,800 | <4,100 | <620 | <6.1 | <4,700 | |
| Titanium | 5.4 | 0.7 | <24 | <30 | <12 | 710 | 2.0 | 720 | |
| Vanadium | 370 | <1.0 | 300 | 670 | 230 | 160 | 1.1 | 390 | 58 |
| Zinc | 9.7 | 70 | 58 | 140 | 51 | 43 | 1.0 | 95 | 68 |

[a] Blanks indicate value not calculated.

## TABLE 5-9. MATERIAL BALANCE FOR METALS, RUN NO. 6

| Metal | Materials to kiln, g/hr Coke | Waste fuel | Limestone feed | Total in | Materials from kiln, g/hr Lime product | Baghouse dust | Stack gas particulates | Total out | Material balance closure[a], % |
|---|---|---|---|---|---|---|---|---|---|
| Aluminum | <140 | <23 | 2,400 | 2,600 | 4,500 | 1,200 | 6.9 | 5,700 | 219 |
| Boron | <23 | 13 | <88 | <120 | <44 | 17 | <0.6 | <62 | |
| Barium | <35 | <6 | <210 | <250 | <110 | 34 | <0.7 | <140 | |
| Beryllium | <1.2 | <0.1 | 28 | 29 | 17 | 3.7 | 0 | 21 | 72 |
| Calcium | <180 | <6.2 | 3,700,000 | 3,700,000 | 3,300,000 | 360,000 | 210 | 3,700,000 | 100 |
| Cadmium | <4.6 | <0.8 | <28 | <33 | <15 | <2.2 | <0.1 | <17 | |
| Cobalt | <6.0 | 10 | <43 | <59 | 32 | 19 | 0.2 | 51 | |
| Chromium | <9.0 | 5.4 | 860 | 870 | 560 | 270 | 2.3 | 830 | 95 |
| Copper | <16 | <2.8 | 590 | 610 | 400 | 69 | <0.4 | 470 | 77 |
| Iron | 140 | 15 | 14,000 | 14,000 | 14,000 | 3,300 | 6.0 | 17,000 | 121 |
| Magnesium | 78 | <0.1 | 2,400,000 | 2,400,000 | 2,100,000 | 230,000 | 140 | 2,300,000 | 96 |
| Mangenese | 2.0 | 1.0 | 1,900 | 1,900 | 1,600 | 170 | 0.3 | 1,800 | 95 |
| Molybdenum | <11 | <1.9 | <66 | <79 | <33 | <5.0 | <0.2 | <38 | |
| Sodium | <6,900 | <1,200 | 58,000 | 66,000 | 34,000 | 5,100 | <4.5 | 39,000 | 59 |
| Nickel | 160 | <3.3 | 580 | 740 | 380 | 130 | 0.7 | 510 | 69 |
| Lead | <76 | 61 | <460 | <600 | <230 | 460 | 1.8 | <690 | |
| Tin | <1,500 | <260 | <9,500 | <12,000 | <4,600 | 690 | <20 | <5,300 | |
| Titanium | <4.4 | 1.1 | <27 | <33 | <380 | 1,800 | 2.8 | <2,200 | |
| Vanadium | <190 | 1.7 | 360 | <550 | 260 | 240 | 1.1 | 500 | |
| Zinc | 6.9 | 110 | 43 | 160 | 93 | 100 | 1.2 | 190 | 119 |

[a] Blanks indicate value not calculated.

TABLE 5-10. MATERIAL BALANCE FOR METALS, RUN NO. 7

| | Materials to kiln, g/hr | | | | Materials from kiln, g/hr | | | | |
|---|---|---|---|---|---|---|---|---|---|
| Metal | Coke | Waste fuel[a] | Limestone feed[a] | Total in | Lime product[a] | Baghouse dust | Stack gas particulates[a] | Total out | Material balance closure[b], % |
| Aluminum | <140 | <8.9 | 2,500 | 2,600 | 1,800 | 710 | 7.6 | 2,500 | 96 |
| Boron | 30 | 5.0 | <85 | <120 | <44 | <6.5 | 1.7 | <52 | |
| Barium | <38 | <2.3 | <210 | <250 | <110 | <16 | 0.7 | <130 | |
| Beryllium | 1.9 | 0 | 28 | 30 | 19 | 3.4 | 0 | 22 | 73 |
| Calcium | 74 | 9.5 | 3,500,000 | 3,500,000 | 3,300,000 | 360,000 | 210 | 3,700,000 | 106 |
| Cadmium | <4.9 | <0.3 | <28 | <33 | <14 | <2.1 | <0.1 | <16 | |
| Cobalt | <6.7 | 4.0 | 36 | <46 | <18 | 7.5 | <0.1 | <26 | |
| Chromium | 7.8 | 2.0 | 870 | 880 | 590 | 150 | 2.0 | 740 | 84 |
| Copper | 17 | <1.0 | 580 | 600 | 360 | 55 | <0.4 | 420 | 70 |
| Iron | 110 | 6.9 | 15,000 | 15,000 | 11,000 | 2,100 | 4.3 | 13,000 | 87 |
| Magnesium | 17 | 0 | 2,400,000 | 2,400,000 | 2,300,000 | 240,000 | 140 | 2,500,000 | 104 |
| Mangenese | 1.6 | 0.4 | 1,800 | 1,800 | 1,400 | 150 | 0.3 | 1,600 | 89 |
| Molybdenum | <12 | <0.7 | <66 | <79 | <32 | <4.9 | <0.2 | <38 | |
| Sodium | <7,300 | <450 | 52,000 | 62,000 | <21,000 | 4,000 | <97 | <25,000 | |
| Nickel | 150 | 1.3 | 580 | 730 | 390 | 120 | 0.7 | 510 | 70 |
| Lead | <81 | 25 | <460 | <570 | <230 | 200 | <1.5 | <430 | |
| Tin | <1,600 | <99 | <9,100 | <11,000 | <4,600 | <690 | <5.4 | <5,300 | |
| Titanium | <4.6 | 0.5 | <26 | <31 | <13 | 580 | 1.2 | 590 | |
| Vanadium | 410 | <0.7 | 360 | 770 | 300 | 220 | 1.3 | 520 | 68 |
| Zinc | 8.7 | 48 | 36 | 93 | 12 | 56 | 1.3 | 69 | 74 |

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[a] Used samples and/or flow rates taken prior to 1100 hours.

[b] Blanks indicate value not calculated.

## TABLE 5-11. MATERIAL BALANCE FOR METALS, RUN NO. 8

| Metal | Materials to kiln, g/hr: Coke | Waste fuel | Limestone feed | Total in | Materials from kiln, g/hr: Lime product | Baghouse dust | Stack gas particulates | Total out | Material balance closure[a], % |
|---|---|---|---|---|---|---|---|---|---|
| Aluminum | <110 | <33 | 3,100 | 3,200 | 3,300 | 800 | 7.6 | 4,100 | 128 |
| Boron | <12 | 9.6 | <92 | <110 | <45 | 9.6 | 1.2 | <56 | |
| Barium | <28 | <8.5 | <220 | <260 | <110 | <17 | 0.8 | <130 | |
| Beryllium | 1.7 | <0.1 | 38 | 40 | 21 | 3.6 | 0 | 25 | 63 |
| Calcium | 100 | 39 | 3,800,000 | 3,800,000 | 3,500,000 | 380,000 | 240 | 3,900,000 | 103 |
| Cadmium | <3.7 | <1.1 | <29 | <34 | <15 | 2.2 | 0.2 | <17 | |
| Cobalt | <4.8 | 15 | <38 | <58 | <19 | 14 | 0.3 | <33 | |
| Chromium | <5.9 | 7.3 | 1,200 | 1,200 | 650 | 200 | 3.4 | 850 | 71 |
| Copper | <13 | 8.2 | 604 | 620 | 400 | 61 | 0.8 | 460 | 74 |
| Iron | 1,200 | 22 | 35,000 | 36,000 | 16,000 | 2,500 | 25 | 19,000 | 53 |
| Magnesium | 70 | <0.1 | 2,600,000 | 2,600,000 | 2,400,000 | 250,000 | 170 | 2,700,000 | 104 |
| Mangenese | 3.7 | 1.5 | 1,900 | 1,900 | 1,700 | 160 | 0.4 | 1,900 | 100 |
| Molybdenum | <8.7 | 2.6 | <69 | <80 | <35 | 5.2 | 0.3 | <41 | |
| Sodium | <5,500 | <1,700 | <44,000 | <51,000 | <22,000 | 4,800 | 320 | <27,000 | |
| Nickel | 120 | 4.7 | 730 | 850 | 420 | 120 | 3.5 | 540 | 64 |
| Lead | <61 | 87 | <480 | <630 | <240 | 360 | 2.1 | <600 | |
| Tin | <1,200 | <370 | <9,600 | <11,000 | <4,800 | <720 | <6.9 | <5,500 | |
| Titanium | <3.4 | 1.6 | <28 | <33 | <14 | 1,100 | 3.0 | <1,100 | |
| Vanadium | 330 | <2.4 | 600 | 930 | 340 | 200 | 1.3 | 540 | 58 |
| Zinc | 5.3 | 170 | 76 | 250 | 56 | 120 | 1.6 | 180 | 72 |

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[a] Blanks indicate value not calculated.

product), although a portion of total emissions of metals exit the kiln in the baghouse dust (10% to 30%). Less than 2% of metals exit the kiln in the stack gas particulates.

As indicated by the average mass balance of 87%, the mass balance was commonly less than 100% (or the mass out was typically less than the mass in). This indicates that a portion of the kiln effluent for metals was not always accounted for by the sampling and analysis techniques.

Metals of greater toxicity, such as barium, cadmium, chromium, and lead, generally were emitted in mass rates near or below the LOQ for the ICAP analytical method.

## 5.4 KILN OPERATION DISCUSSION

The following subsections discuss the results of sampling and analyses with respect to the operation of the kiln as well as various operation problems encountered during the test burn, the causes of such problems, and, if applicable, their solutions.

### 5.4.1 CO Emissions vs. DRE

As indicated in Section 4.2.5, the CO emissions fluctuated widely each day. High peaks of CO indicate incomplete combustion or kiln "upset" conditions. Samples for organics (VOST) and particulates (EPA Method 5) were taken at varying kiln conditions or CO emission rates as shown in Figures A-1 through A-8 in Appendix A. As shown by the organic emission results (Table 5-1), destruction and removal efficiencies (DRE) did not change as the kiln operation changed. It is important to note that at non-optimum kiln conditions (high CO emissions) high DREs were obtained (>99.99%). In addition, other pollutant emission, such as particulates and HCl, did not vary under the high CO emission conditions.

### 5.4.2 Causes for CO Emission Fluctuations

Causes for the wide fluctuations in CO emissions are numerous. Clumps of petroleum coke periodically were fed into the kiln due to a wet supply of coke. This caused excess fuel conditions, which resulted in excess $SO_2$ and CO emissions (incomplete combustion). For example, this can be observed in Figure A-5 in Appendix A for Day 5 when $SO_2$ spikes are followed by CO emission spikes.

Another cause for CO fluctuations is a non-constant fuel rate (whether coke, gas, and/or waste fuel). Although the fuel systems are set up for constant feed rates, variations in feed rates inevitably occur, causing excess fuel or air conditions in the kiln, which, in turn, result in variable CO emissions.

CO fluctuation can also be caused by product rushes. A rush is a flood or wave of hot powder of product (the powder is the lime product than has not yet reached its final form) to the combustion zone in the kiln. This wave of hot powder interferes with combustion; uncombusted material is released and read as high CO emissions.

CO fluctuations can also be caused by the general operation of the kiln. Unfortunately, waste fuel was burned only 8 hours per day, whereas, at least 24 hours of burning is needed to allow "fine-tuning" of the kiln to minimize the minor types of fluctuations that caused varying CO emissions.

### 5.4.3 $NO_x$ /$SO_2$ Emissions vs. Flame Conditions

Based on the kiln flame theory with respect to $NO_x$ and $SO_2$ emissions (discussed in Section 2.3), one objective during the test burn was to maximize the $NO_x$ and minimize the $SO_2$ emissions which, ideally, would produce the hottest or most intense flame and provide the best conditions for complete destruction of organics. Proof of this theory for this particular test burn would have been evident if maximum DREs had been obtained at maximum $NO_x$/minimum $SO_2$ emissions and if lower DREs had been obtained as the $NO_x$ was reduced and $SO_2$ increased. Unfortunately, proof of this concept was not possible in this test for two reasons. First, little change was found in DRE values, as high DREs were obtained at all kiln conditions (i.e., any $NO_x/SO_2$ emission rates). Second, fine-tuned operation of the kiln that could control and maintain $NO_x$ and $SO_2$ emissions at desired levels was not possible. Figures A-1 through A-8 in Appendix A show this second point in graphical form. Although a wide variety of $SO_2$ and $NO_x$ concentrations were monitored, no correlation between $NO_x/SO_2$ could be defined nor were constant $NO_x/SO_2$ concentrations maintained for long periods of time.

### 5.4.4 Burner System

The temporary waste fuel burner setup, although sufficient for the test burn, did not allow optimum mixing of coke and waste fuel. As shown in Figure 2-6, the waste fuel pipe was laid on top of the main burner, and waste fuel was injected into the flame. However, the coke and primary air exited the burner most likely in a spray-type manner, which made it difficult for the waste fuel to mix well with the coke and air as shown in Figure 5-2. This became obvious at low waste fuel feed rates in which puffing of the flame occurred due to an insufficient amount of waste fuel mixed with the coke to maintain the flame. This situation did not necessarily indicate that the waste fuel was not completely combusted as indicated by high DREs but rather that the waste fuel could be more efficiently utilized to maintain the flame, especially at low rates.

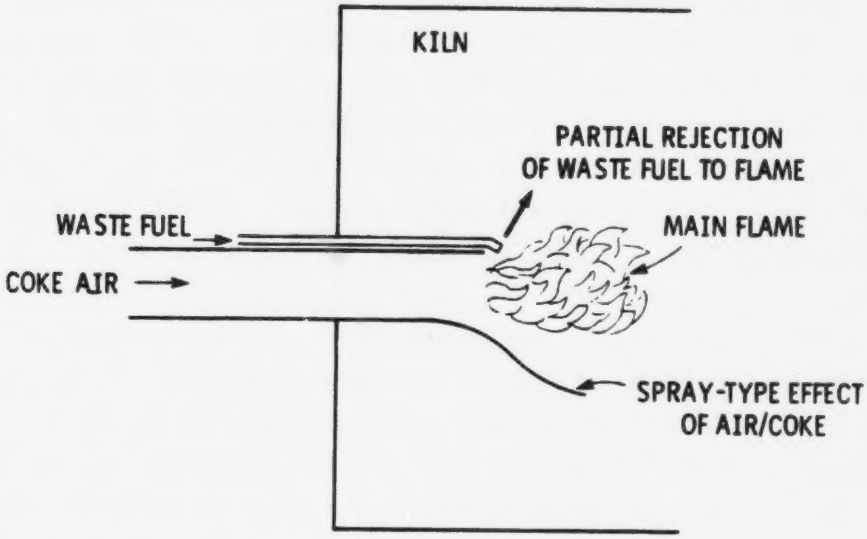
KILN
PARTIAL REJECTION OF WASTE FUEL TO FLAME
WASTE FUEL
MAIN FLAME
COKE AIR
SPRAY-TYPE EFFECT OF AIR/COKE

Figure 5-2. Example of coke/waste fuel burner system.

### 5.4.5 Product Quality Problems

Under normal conditions, the lime kiln operators maintain the $O_2$ content in the kiln at low levels (~1.5%). However, after the switch was made from gas to waste fuel, consistent kiln operation could not be maintained; CO emissions fluctuated as previously discussed; $O_2$ levels increased; and on a few occasions, lime product quality problems were encountered. The following discussion provides possible explanations for the causes of this problem.

Theoretically, low sulfur content in the product is one requirement for producing a good quality lime product. To obtain low sulfur in the product, high sulfur (or $SO_2$) stack emissions must result. However, as noted in the flame theory discussed in Section 2.3, localized reducing conditions in the flame (a non-optimum flame) create high-$SO_2$ stack emissions. Thus, to produce a good quality low-sulfur lime product, the kiln must maintain localized reducing conditions and sacrifice flame optimization.

As the waste fuel was fed to the kiln during the test burn, however, the localized reducing condition in the flame decreased, increasing the $O_2$ in the kiln, lowering $SO_2$ stack gas emission, and increasing the sulfur in the lime product. The flame localized condition decreased because the waste fuel, being a highly volatile material, burned quicker than the gas or coke and produced a more intense flame, thereby optimizing the flame. This flame provided more intense heat to the kiln, increasing temperatures and requiring less $O_2$ to burn the waste fuel. Unfortunately, as described above, this optimum flame resulted in higher sulfur in the lime product.

If waste fuel is to be burned, the follow conditions should make it possible to maintain an optimum flame while reducing $O_2$ content in the kiln and reducing sulfur in the lime product:

- Increase the limestone feed rate gradually, thus, allowing the operator sufficient time (>24 hrs) to equilibrate the kiln for temperature and $O_2$ because a more intense flame is being realized due to the waste fuel being used.

- Decrease ID fan speed (i.e., reduce the draft) to lower the $O_2$ in the kiln resulting in lower sulfur in product and higher sulfur in the stack. This option could not be performed during the test burn because draft rates were already at a minimum. However, it is an option available to the site for future reference.

## 5.5 BASELINE VS. WASTE FUEL CONDITIONS

In addition to determining pollutant emissions, calculating destruction efficiencies, and determining the fate of chlorine and metals, the test burn also compared pollutant emissions under baseline and waste fuel conditions.

Statistical methods are available to aid in the study of a change in conditions. The fairly simple "t-test" can be used to determine whether a statistically significant difference exists between one set of data and another at a selected degree of certainty (assuming the populations are approximately normal) [6, 7].

Table 5-12 shows a statistical comparison for particulate, HCl, $NO_x$, $SO_2$, CO, total hydrocarbons, and POHCs for baseline and waste fuel conditions. According to Table 5-12, HCl, total hydrocarbons, methylene chloride, and toluene had a significant difference from the baseline to the waste fuel conditions. All other pollutant concentrations did not change from baseline to waste fuel conditions. The use of a 95% certainty level indicates that this conclusion has only a 5% chance of being incorrect.

Only HCl increased due to waste fuel firing. Total hydrocarbons actually decreased as natural gas was cut-off and waste fuel was fired in the kiln. For POHCs, only methylene chloride and toluene increased to levels significantly different than baseline conditions. However, as shown previously in Table 5-1, $MeCl_2$ and toluene were still destroyed by >99.99% during waste fuel conditions.

It should be noted that the pollutant concentrations listed in Table 5-12 are averages of all samples taken under various waste fuel feed rates and kiln operating conditions (ranging from stable to unstable) and that the analysis of data by separate runs was not performed.

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[6] Walpole, R. E and R. H. Myers, Probability and Statistics for Engineers and Scientists. MacMillan Company, New York, NY, 1972. pp. 192-201.

[7] Bennett, C. A. and N. C. Franklin, Statistical Analysis in Chemistry and the Chemical Industry. John Wiley & Sons, Inc., New York, NY, 1954. pp. 176-180.

TABLE 5-12. COMPARISON OF POLLUTANT EMISSIONS UNDER BASELINE AND WASTE FUEL CONDITIONS

| Pollutant | Mean loading | | Statistical difference with 95% confidence |
|---|---|---|---|
| | Baseline | Waste fuel | |
| Particulate | 28.7 ± 4.7 $mg/m^3$<br>(n = 4) | 35.3 ± 8.0 $mg/m^3$<br>(n = 8) | No statistical difference |
| HCl | 3.1 ± 4.0 $mg/m^3$<br>(n = 4) | 7.0 ± 1.7 $mg/m^3$<br>(n = 8) | Statistically different |
| $NO_x$[a] | 386 ± 49 ppm<br>(n = 47) | 446 ± 64 ppm<br>(n = 160) | Statistically different |
| $SO_2$[a] | 553 ± 110 ppm<br>(n = 25) | 596 ± 240 ppm<br>(n = 160) | No statistical difference |
| CO[a] | 477 ± 966 ppm<br>(n = 68) | 599 ± 1,409 ppm<br>(n = 165) | No statistical difference |
| Total hydrocarbons[a] | 8.2 ± 1.9 ppm<br>(n = 13) | 3.5 ± 1.1 ppm<br>(n = 143) | Statistically different |
| POHCs | | | |
| Methylene chloride | $5 \times 10^{-5} \pm 11 \times 10^{-5}$ g/min<br>(n = 17) | $14 \times 10^{-5} \pm 37 \times 10^{-5}$ g/min<br>(n = 27) | Statistically different |

(continued)

TABLE 5-12 (continued)

| Pollutant | Mean loading |  | Statistical difference with 95% confidence |
|---|---|---|---|
|  | Baseline | Waste fuel |  |
| POHCs (continued) |  |  |  |
| Methyl ethyl ketone | $42 \times 10^{-5} \pm 56 \times 10^{-5}$ g/min<br>(n = 17) | $63 \times 10^{-5} \pm 104 \times 10^{-5}$ g/min<br>(n = 28) | No statistical difference |
| 1,1,1-Trichloroethane | $29 \times 10^{-5} \pm 76 \times 10^{-5}$ g/min<br>(n = 17) | $44 \times 10^{-5} \pm 92 \times 10^{-5}$ g/min<br>(n = 28) | No statistical difference |
| Trichloroethylene | $16 \times 10^{-5} \pm 28 \times 10^{-5}$ g/min<br>(n = 16) | $19 \times 10^{-5} \pm 38 \times 10^{-5}$ g/min<br>(n = 28) | No statistical difference |
| Tetrachloroethylene | $17 \times 10^{-5} \pm 14 \times 10^{-5}$ g/min<br>(n = 17) | $26 \times 10^{-5} \pm 60 \times 10^{-5}$ g/min<br>(n = 28) | No statistical difference |
| Toluene | $100 \times 10^{-5} \pm 120 \times 10^{-5}$ g/min<br>(n = 17) | $199 \times 10^{-5} \pm 364 \times 10^{-5}$ g/min<br>(n = 28) | Statistically different |

[a] Continuous analysis; mean loadings determined by average of 15-minute averages.

# SECTION 6

# CONCLUSIONS

This test burn was completed to (1) conduct sampling and analysis during the burning of liquid hazardous organic waste as supplemental fuel; (2) calculate the DRE for each POHC; (3) determine the concentrations of particulate matter, HCl, $SO_2$, $NO_x$, CO and total hydrocarbon in the stack gas; (4) determine the fate of chlorine and trace metals in stack gas and process samples; (5) compare baseline pollutant emisssions to those for waste fuel conditions; (6) evaluate the kiln performance and DREs with respect to $SO_2$, $NO_x$, and/or CO emissions; and (7) evaluate the kiln operation during waste fuel conditions with respect to quality product generation. Many of these objectives were met as described below:

- Consistent achievement of at least 99.99% destruction and removal efficiency was demonstrated for each POHC (methylene chloride, methyl ethyl ketone, 1,1,1-trichloroethane, trichloroethylene, tetrachloroethylene, and toluene) during each test day. DREs for each POHC ranged and averaged as follows:

| POHC | Range, % | Average, % |
|---|---|---|
| $MeCl_2$ | 99.598 to 99.999 | 99.997 |
| MEK | 99.998 to 99.999 | 99.999 |
| $CH_3Cl_3$ | 99.989 to 99.999 | 99.997 |
| TCE | ≧99.999 | 99.999 |
| Perc | ≧99.999 | 99.999 |
| Toluene | ≧99.999 | 99.999 |

Only four runs were less than 99.99%, three for methylene chloride, and one for 1,1,1-trichloroethane. The $MeCl_2$ results should be reviewed with caution as the potential for laboratory contamination is high for this material.

- Emissions of pollutants in the stack gas were determined and ranged as follows:

| Pollutant | Condition | Range | | |
|---|---|---|---|---|
| | | kg/hr | ppm | lb/hr |
| Particulate | Baseline | 0.66-1.1 | | 1.5-2.5 |
| | Waste fuel | 0.68-1.4 | | 1.5-6.0 |
| HCl | Baseline | 0.04-0.17 | | 0.08-0.37 |
| | Waste fuel | 0.12-0.26 | | 0.25-0.58 |
| $SO_2$ | Baseline | | 123-730 | |
| | Waste fuel | | 180-2,100 | |
| $NO_x$ | Baseline | | 300-460 | |
| | Waste fuel | | 280-550 | |
| CO | Baseline | | 10-5,000 | |
| | Waste fuel | | 10-5,000 | |
| THC | Baseline | | 6-13 | |
| | Waste fuel | | 1.5-10 | |

Particulate and HCl emissions were low, as expected, compared to other test burns and typical lime kiln facilities. Wide variability occurred in CO emissions and, on occasion, in $SO_2$ emissions due to changes made in the kiln operation. THC concentrations were consistently low (<13 ppm).

- Greater than approximately 90% of the chlorine introduced into the kiln came from the limestone feed; less than 4% of the chlorine came from the waste fuel. Approximately 50% of the chlorine left the kiln system in the lime product; 50% left in the baghouse dust. Less than 5% of the total chlorine exited through the stack.

- Approximately 80 to 100% of the total sulfur is introduced to the kiln in the coke feed and very little is introduced in the waste fuel (<0.5%). The sulfur exits the kiln in the following streams and proportions: stack gas (50 to 80%), baghouse dust (15-35%), and lime product (2 to 15%).

- Typically, more than 90% of the trace metals are introduced into the kiln by the limestone feed. Coke and waste fuel contributed a very small portion of the metals to the kiln. Only zinc had a higher contribution (~50%) by the waste fuel feed and vanadium had a higher contribution (~55%) by coke feed. The trace metals generally exit the kiln system in the lime product (70% to 90% of total emissions), although a portion of the total emissions exit the kiln in the baghouse dust (10% to 25%). Metals of higher toxicity (e.g., barium, cadmium, chromium, and lead) generally were emitted in rates near of below the limit of quantification for the analytical method used.

- A statistical comparison was conducted of stack gas emissions at baseline vs. waste fuel conditions. Particulate, $SO_2$, CO, and four POHCs (MEK, $CH_3CCl_3$, TCE, and Perc) showed no statistical difference in emissions from baseline to waste fuel conditions. HCl and $NO_x$ showed an increase in emissions from baseline to waste fuel conditions while total hydrocarbons actually decreased in emissions. Al though $MeCl_2$ and toluene emissions increased from baseline to waste fuel conditions, DREs remained above 99.99% for all conditions.

- A correlation between $SO_2/NO_x$ emissions was desired, but due to wide fluctuation in kiln operation (as noted by fluctuating CO emissions), no $SO_2/NO_x$ correlation can be made. In addition, VOST samples to determine DREs for organics were taken at low, medium, and high CO emissions (variable kiln conditions), and results show that high DREs (>99.99%) were achieved at virtually any CO emission rate.

- During waste fuel testing, the kiln had problems with lime product handling thought to be caused by excess chlorine from the waste fuel or excess sulfur resulting from nonstable kiln operation (based on CO emissions data and changes in kiln parameters made hourly by operators). Causes for this are uncertain but most likely include the following: (1) the waste fuel, being a highly volatile material, burned quicker than coke or gas and produced a more intense flame, thereby optimizing the flame, decreasing localized reducing conditions in the flame, increasing the $O_2$ in the kiln, lowering $SO_2$ stack gas emissions, and increasing the sulfur in the lime poduct; (2) a better burner system needs to be installed such that the waste fuel and coke are mixed well; or (3) operators were inexperienced with new fuel and did not have sufficient "practice" time using a new fuel supply/system to provide stable operation.

Possible solutions to the operational problems may include: (1) increase the limestone feed gradually allowing the operator sufficient time to equilibrate the kiln for temperature, $O_2$, and a quality lime product; and (2) decrease ID fan speed (i.e., reduce the draft) to lower the $O_2$ in the kiln, resulting in higher sulfur in the stack and lower sulfur in the product.

# SECTION 7

# QA/QC RESULTS

Quality assurance/quality control goals for precision and accuracy were set prior to initiating the sampling and analyses at Rockwell Lime Company as shown in Tables 7-1 and 7-2 (also see Reference 1 listed in Section 3.3). The QA plan was reviewed and commented on by IERL-Cincinnati's QA contractor. The following sections describe QA/QC results as compared to program goals.

## 7.1 QA/QC RESULTS FOR STACK GAS SAMPLING AND ANALYSIS

Stack gas samples were taken and analyzed for particulates, HCl, metals, organics, carbon dioxde, oxygen, sulfur dioxide, nitrogen oxides, carbon monoxide, and total hydrocarbons. Table 7-3 summarizes QA/QC results for all of the stack gas samples, except organics (POHCs) and metals, which are presented in Sections 7.2 and 7.5, respectively.

Sampling and analysis completeness goals were met or exceeded for all parameters listed in Table 7-3. All other goals were met except percent relative difference for HCl was exceeded, a missing spike sample for HCl, and one less sample was taken than the goal for continuous $SO_2$ data. The goals met included the number of blanks taken, the number of splits performed and the percent relative difference, the number of standards analyzed and the percent recovery, and the number of standard calibrations performed on the continuous monitor each day.

TABLE 7-1. QA OBJECTIVES FOR PRECISION FOR EACH MAJOR MEASUREMENT

| Measurement parameter (method) | No. of samples | No. of blanks | No. of splits | Precision goals, relative difference, % | | S&A completeness goals, % |
|---|---|---|---|---|---|---|
| | | | | Within laboratory | Between laboratory | |
| 1 Principal organic hazardous constituents (VOST,GC/MS) | 42 pairs of sorbent tubes | 10[a] | 1[b] | 25[b] | NA | 95 |
| 2 Particulate matter (EPA Method 5) | 8 | 2 | NA | NA | NA | 100 |
| 3 Hydrogen chloride (absorption, SIE) | 8 | 2 | 2 | 10 | NA | 100 |
| 4 Carbon dioxide and oxygen (EPA Method 3) | 24 | NA | 8[c] | 7.5 for $CO_2$<br>2.0 for $O_2$ | NA | 100 |
| 5 Nitrogen oxides (Chemiluminescence) | 7 | NA | NA | NA | NA | 100 |
| 6 Sulfur dioxide (fluorescence) | 7 | NA | NA | NA | NA | 100 |
| 7 Carbon monoxide (EPA Method 10) | 7 | NA | NA | NA | NA | 100 |
| 8 Total hydrocarbons (FID) | 7 | NA | NA | NA | NA | 100 |
| 9 POHC's in waste fuel (grab → composite, GC/MS) | 5 | 1 | 1 | 10 | NA | 100 |

(continued)

TABLE 7-1 (continued)

| Measurement parameter (method) | No. of samples | No. of blanks | No. of splits | Precision goals, relative difference, % |  | S&A completeness goals, % |
|---|---|---|---|---|---|---|
|  |  |  |  | Within laboratory | Between laboratory |  |
| 10 Metals in particulate matter, waste fuel, coal/coke, lime product, dry limestone feed, and baghouse dust (ICP) | 41[d] | 6 | 6 | 15 | NA | 100 |
| 11 Chlorine and sulfur in waste fuel, coal/coke, lime product, dry limestone feed, and baghouse dust (XRF) | 33[e] | 5 | 5 | 10 | NA | 100 |
| 12 Btu and ash in waste fuel and coal/coke | 14 | 2 | 2 | 10 | NA | 100 |

---

[a] Two field blank pairs (4), two trip blanks (2), two lab blanks (2), and two baseline lab blanks (2).

[b] This will be the only "sampling and analytical" split run in the program; a simultaneous side-by-side sample run will be conducted. All other splits refer to "analytical" splits only.

[c] EPA Method 3 is run in triplicate until the results of any three analyses differ by no more than the percent volume stated in the reference method.

[d] Five waste fuel samples, eight particulate filters, and seven of the remaining matrices.

[e] Five waste fuel samples and seven of the remaining matrices.

NA = Not applicable.

## TABLE 7-2. QA OBJECTIVES FOR ACCURACY FOR EACH MAJOR MEASUREMENT

| Measurement parameter (method) | No. of samples | No. of spikes | Reference standards | Accuracy goals, percent recovery | |
|---|---|---|---|---|---|
| | | | | Spikes | Reference standards |
| 1 Principal organic hazardous constituents tuents (VOST, GC/MS) | 42 pairs of sorbent tubes | 42 to 84[a] | 3 x 3[b] | NA | -[c] |
| 2 Particulate matter (EPA Method 5) | 8 | NA | -[d] | NA | ±5 of calibration factor for dry gas meter, ±0.2 for analytical balance |
| 3 Hydrogen chloride (absorption, SIE) | 8 | 1 | 5[e] | NA | NA[e] |
| 4 $CO_2$ and $O_2$ (EPA Method 3) | 24 | NA | 4[f] | NA | ±5 |
| 5 Nitrogen oxides (chemiluminescence) | 7 | NA | 7 | NA | ±2 |
| 6 Sulfur dioxide (fluorescence) | 7 | NA | 7 | NA | ±2 |
| 7 Carbon monoxide (EPA Method 10) | 7 | NA | 7 | NA | ±2 |
| 8 Total hydrocarbons (FID) | 7 | NA | 7 | NA | ±2 |
| 9 POHCs in waste fuel (grab + composite, GC/MS) | 5 | 7[c] | -[g] | NA | -[c] |
| 10 Metals in particulate matter, waste fuel, coal/coke, lime product, dry limestone feed, and baghouse dust (ICP) | 41[d] | 6 x 21[h] | 6 x 21[h] | ±20 (80-120%) | ±10 |
| 11 Chlorine and sulfur in waste fuel, coal/coke, lime product, dry limestone feed, and baghouse dust (XRE) | 33 | 3 | 6 | ±10 | ±10 |
| 12 Btu and ash in waste fuel and coal/coke | 14 | 1 | 1 | ±10 | ±10 |

[a] Each sorbent tube is spiked with an internal standard. The first analyses will be conducted on the front tube and back-up tube. If the gas volumes samples are sufficiently small such that breakthrough of the front tube does not occur, then the remaining back-up tubes will not be analyzed.

[b] Refers to each of three POHC external standards run at three levels bracketing the amount captured for sorbent tube.

[c] Rather than reference standards, external standards of each POHC are used for quantitation.

[d] Reference standards for particulate matter do not exist. EPA Method 5 is considered "accurate" if the dry gas meter, stack and meter temperature sensors, probe nozzle, and analytical balance demonstrate acceptable calibration.

[e] A five-point standards calibration curve is established for analysis.

[f] Bottled gases traceable to NBS containing 2-4% $O_2$, 15% $O_2$, 2-4% $CO_2$, and 14-18% $CO_2$.

[g] Each constituent >1% in the waste fuel is quantified by a separate external standard of the same chemical structure. All other constituents <1% in the waste are semi-quantified by comparison with an external standard which elutes in the vicinity of that constituent.

[h] Twenty-one different elements are spiked and have reference standards run.

## TABLE 7-3. QA/QC RESULTS FOR STACK GAS SAMPLES

| Parameter and units | Split samples |  |  |  | Standard samples |  |  | Spiked samples[a] | Completeness goals, % (No. of actual samples/goals) | No. of blanks (field and lab) |
|---|---|---|---|---|---|---|---|---|---|---|
|  | Result 1 | Result 2 | Average | Relative difference, % | Observed | Actual | Recovery, % |  |  |  |
| Particulate | NA[b] | NA[b] | NA[b] | NA[b] | NA[b] | NA[b] | NA[b] | NA[b] | 125 (10/8) | 5 |
| HCl, µg | 35.0 | 30.0 | 32.5 | 15.4 | 33.0 | 0.0 | 133.0 | NP[c] | 125 (10/8) | 2 |
|  | 1,120.0 | 874.0 | 997.0 | 25.0 | 10.2 | 10.6 | 96.2 |  |  |  |
|  |  |  |  |  | 35.8 | 35.4 | 101.0 |  |  |  |
| $CO_2$ and $O_2$,[d] molecular weight | 32.36 | 32.34 | 32.22 | 0.43 | NA[e] | NA[e] | NA[e] | NA | 150 (36/24) | 0 |
|  | 32.22 | 32.22 | 32.22 | 0.0 |  |  |  |  |  |  |
|  | 32.12 | 32.26 | 32.20 | 0.43 |  |  |  |  |  |  |
|  | 31.54 | 31.54 | 31.52 | 0.06 |  |  |  |  |  |  |
|  | 31.80 | 31.72 | 31.66 | 0.44 |  |  |  |  |  |  |
|  | 31.52 | 31.58 | 31.76 | 0.76 |  |  |  |  |  |  |
|  | 31.84 | 31.72 | 31.70 | 0.44 |  |  |  |  |  |  |
|  | 31.68 | 31.66 | 31.62 | 0.25 |  |  |  |  |  |  |
|  | 31.80 | 31.86 | 31.74 | 0.38 |  |  |  |  |  |  |
|  | 31.68 | 31.68 | 31.66 | 0.06 |  |  |  |  |  |  |
|  | 31.58 | 31.74 | 31.56 | 0.57 |  |  |  |  |  |  |
|  | 31.64 | 31.62 | 31.54 | 0.32 |  |  |  |  |  |  |
| $SO_2$, continuous | NA | NA | NA | NA | Standard calibration gases used three to four times per day at zero, low, medium, and high scale ranges |  |  | NA | 86 (6/7) | NA |
| $NO_x$, continuous | NA | NA | NA | NA |  |  |  | NA | 88 (7/8) |  |
| $O_2$, continuous | NA | NA | NA | NA |  |  |  | NA | 86 (6/7) |  |
| THC, continuous | NA | NA | NA | NA |  |  |  | NA | 75 (6/8) |  |
| CO, continuous | NA | NA | NA | NA |  |  |  | NA | 100 (8/8) |  |
| $SO_2$ (EPA Method 6) mg/dscm | 1,152.0 | 1,146.0 | 1,149.0 | 0.5 | 1,149.0 | 1,144.0 | 101.0 | NA | 300 (6/2) | 2 |
|  |  |  |  |  | 2,297.0 | 2,288.0 | 100.0 |  |  |  |
| $SO_2$ (EPA Method 6 versus cont.), ppm | 535.0 | 587.0[f] | 561.0 | 9.3 |  |  |  |  |  |  |
|  | 628.0 | 589.0[f] | 609.0 | 6.4 |  |  |  |  |  |  |
|  | 603.0 | 546.0[f] | 575.0 | 9.9 |  |  |  |  |  |  |
| $NO_x$ (EPA Method 7), µg | 913.0 | 922.0 | 917.5 | 0.01 | 922.0 | 896.0 | 103.0 | NA | - (4/0) | 0 |
|  | 913.0 | 931.0 | 922.0 | 0.02 | 365.0 | 378.0 | 97.0 |  |  |  |

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[a] No spiked samples analyzed for any samples.

[b] NA = Not applicable; QA/QC for EPA Method 5 includes calibration of sampling equipment and field blanks.

[c] NP = Not performed, although scheduled in Table 7-2.

[d] $CO_2$ and $O_2$ analyzed in triplicate.

[e] Bottled gases used to calibrate Fyrite prior to field use.

[f] Continuous $SO_2$ result over EPA Method 6 sampling period.

## 7.2 QA/QC RESULTS FOR POHC STACK GAS SAMPLING AND ANALYSIS

Quantification of the POHCs in the stack gas was achieved by VOST sampling using Tenax adsobent and thermal desorption GC/MS analysis. Of the QA/QC goals specified in Tables 7-1 and 7-2 for POHC stack gas analysis, only the within-laboratory precision goals for a duplicate sample could not be met. Table 7-4 summarizes QA/QC results for POHC sampling and analysis.

Forty-five pairs of VOST samples were collected and returned to the Dayton Laboratory; however, only forty-one completed pairs were analyzed because several tubes were lost during analyses due to instrument problems. The completeness goal of forty-two samples was nearly met because completed sampling and analyses was 98% (41/42).

One "within-lab" duplicate sample was taken, in which two side-by-side VOST runs were made in the stack (Run Nos. 6C and 6D). Unfortunately, the front tube on Run No. 6C was not analyzed, and comparison of these two runs was not possible.

The number of blanks taken and analyzed exceeded the goal of ten shown in Table 7-1. Fourteen blanks were taken to determine if contamination had occurred, and, if so, when the contamination occurred during sampling and analyses. As shown by blank tube results reported previously in Table 4-10, little contamination occurred during transport (trip blanks) or in the field tubes (field blanks) that would result in tube mass values above the background or baseline levels for any of the POHCs. However, several lab blanks indicate that the possibility of lab contamination on selected samples exists. For example, one of the four lab blanks for 1,1,1-trichloroethane was 57 ng, which is 5 to 15 times greater than the other lab blanks. In addition, methylene

## TABLE 7-4. QA/QC RESULTS FOR POHC SAMPLING AND ANALYSIS

| Duplicate results | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| No. of duplicates | Run Nos. | POHC | Result, $W_{out}$ g/min x $1^{-5}$ | | Relative difference, %[b] | No. of blank samples | No. of spiked samples | No. of standard samples | Completeness goals, % (No. of actual samples taken and analyzed/ goals) |
| | | | 6C[a] | 6D | | | | | |
| 1 (Within lab) | 6C and 6D | Me $Cl_2$ | 16.37 | 1.80 | NA | 6[c] | 83[d] | 3 x 6[e] | 98 (41/42) |
| | | MEK | 26.19 | 37.55 | NA | | | | |
| | | $CH_3CCl_3$ | 158.47 | 10.55 | NA | 8[f] | | | |
| | | TCE | 48.51 | 8.86 | NA | | | | |
| | | Perc | 6.92 | 7.02 | NA | | | | |
| | | Toluene | 45.24 | 186.76 | NA | | | | |

[a] Back tube only; front tube not analyzed.

[b] Not calculated as Run 6C had back tube only.

[c] Includes two lab, two field, and two trip for TCE and Perc only.

[d] Each tube spiked with 10 ng of benzene-$d_6$ internal standard.

[e] Standards for each POHC run at three levels.

[f] Includes four lab, two field, and two trip for all POHCs.

chloride is commonly used in the lab as a solvent and the possibility of sample contamination by that compound is high even though blanks did not detect any contamination. The fact that contamination could have occurred on several samples did not affect the DRE results, however, as all but 4 runs were greater than 99.99% DRE.

Each tube was spiked prior to analysis with 10 ng of benzene-$d_6$ internal standard. The internal standard was monitored during the analysis to check GC/MS performance. Eighty-three tube samples were analyzed, excluding blanks; this included forty-two back tubes and forty-one front tubes. Several tubes were not analyzed due to analytical problems (three back and four front tubes).

Six external standards, one for each POHC, were analyzed by GC/MS to determine the correct ion ratios and compound retention times for quantification of each POHC. Standards were prepared at three concentration levels by spiking the standard solutions of the analytes (POHCs) in methanol onto blank tubes and then desorbing the tubes in the same manner as that used for the samples. A three-point calibration curve was established for each POHC which correlated response/ion ratios with concentrations. A new external standard for each POHC was prepared and analyzed daily to verify that the GC/MS system maintained operation on the calibration curve.

## 7.3 QA/QC RESULTS FOR WASTE FUEL

Daily composite samples were collected and returned to the MRC Dayton Laboratory for analysis of POHCs and other organics by capillary GC/MS. All of the samples were analyzed for the six POHCs. Two additional samples from Days 4 and 7 were analyzed for the top twenty compounds. All samples were prescreened to determine the major components and their approximate concentrations so that standards could be prepared at approximately 2X, 1X, and 0.5X of the sample concentrations for POHCs and other major components. Samples were quantified using major ion areas of external standards with correcton for internal standards where possible.

Samples were diluted 1:10 in either of two solvents, nonane (a heavier solvent for the analysis of low-boilding components) or acetone (a lighter solvent for the analysis of the higher molecular weight components). Each sample was analyzed in duplicate for a total of four analyses per sample.

QA/QC results for waste fuel analyses are summarized in Table 7-5. All QA/QC goals for waste fuel sampling and analyses were met, including the number of analytical splits made and percent relative difference, the number of field splits made (1) and the percent relative difference, the number of standards and blanks taken and analyzed, and the completeness goals. Split results for both GC/MS wide-scan and POHC analyses showed excellent relative difference. Results for split samples on individual compounds for wide-scan and POHC are presented in Table B-1 of Appendix B.

Standards are used by the GC/MS system for quantifying compound concentrations, and results are not calculated for percent recovery. The quantifying compounds and ions used are listed in Table B -1 of Appendix B.

## 7.4 QA/QC RESULTS FOR CHLORINE, SULFUR, Btu, AND ASH IN PROCESS SAMPLES

Table 7-6 provides the QA/QC results of the analyses for chlorine, sulfur, Btu, and ash in various process samples. The split samples were duplicates taken in the field. All spikes were prepared and spiked in the lab; no field spikes were used.

## TABLE 7-5. QA/QC RESULTS FOR WASTE FUEL ANALYSES

| No. of samples (parameter) | Split sample results relative difference, % | | | Standard sample results | | No. of blank samples | Completeness goals, % (No. of actual samples/goals) |
|---|---|---|---|---|---|---|---|
| | Range | Average | Standard deviation | No. of samples | Recovery, % | | |
| 46 (Wide scan by GC/MS) | 0.4-27.7 | 9.4 | 7.0 | 5 | NA[a] | 1[b] | 100 (2/2) |
| 30 (POHCs by GC/MS) | 0.4-25.4 | 11.7 | 7.1 | 5 | NA[a] | 1[b] | 100 (5/5) |

[a] Not applicable; each constituent is quantified by an external standard of the same chemical structure; percent recovery is not calculated.

[b] Solvent blank.

## TABLE 7-6. QA/QC RESULTS FOR CHLORINE, SULFUR, Btu, AND ASH

| | | Split sample results | | | Standard or spike sample results | | | | | Completeness goals, % (No. of samples analyzed/goals)[b] |
|---|---|---|---|---|---|---|---|---|---|---|
| | | Results | | | | | | | | |
| Parameter | Units | 1 | 2 | Relative difference, % | Units | Observed value | Actual value or (spiked values) | Sample value[a] | Recovery, % | |
| Chloride | % | 2.93 | 3.15 | 7.2 | % | 0.140 | (0.025) | 0.105 | 140 | 130 (43/33) |
| | % | 0.053 | 0.040 | 28.0 | | 0.108 | (0.025) | 0.080 | 112 | |
| | % | 0.095 | 0.075 | 23.5 | | 0.093 | (0.025) | 0.085 | 32 | |
| | % | 0.140 | 0.125 | 11.3 | | 0.208 | (0.025) | 0.175 | 132 | |
| | % | 1.550 | 1.175 | 27.5 | | 0.175 | (0.025) | 0.140 | 140 | |
| | | | | | | 0.155 | (0.025) | 0.125 | 120 | |
| | | | | | | 0.113 | (0.025) | 0.075 | 152 | |
| | | | | | | 0.790 | (0.025) | 0.750 | 160 | |
| | | | | | | 1.210 | (0.025) | 1.175 | 140 | |
| | | | | | | 1.580 | (0.025) | 1.550 | 120 | |
| | | | | | | 0.832 | (0.025) | 0.800 | 128 | |
| | | | | | | 0.311 | (0.236) | 0.071 | 102 | |
| | | | | | | 0.270 | (0.237) | 0.028 | 102 | |
| | | | | | | 0.285 | (0.253) | 0.400 | 97 | |
| | | | | | | 3.31 | (0.232) | 3.14 | 73 | |
| | | | | | | 3.19 | (0.226) | 3.05 | 62 | |
| Sulfur | % | 0.09 | 0.04 | 76.9 | % | 2.89 | 2.90 | NA[a] | 99.7 | 106 (35/33) |
| | % | 4.14 | 4.08 | 1.5 | | 0.66 | 0.63 | | 105 | |
| | % | 0.12 | 0.08 | 40.0 | | 1.00 | 1.00 | | 100 | |
| | % | 1.36 | 1.31 | 3.7 | | 1.99 | 2.00 | | 99.5 | |
| | | | | | | 3.00 | 3.00 | | 100 | |
| | | | | | | 0.30 | 0.30 | | 100 | |
| | | | | | | 0.126 | 0.124 | | 102 | |
| Btu/lb | | 12,301 | 12,232 | 0.6 | | 11,357.0 | 11,373.0 | | 99.9 | |
| | | 14,255 | 14,174 | 0.6 | | 11,391.0 | 11,373.0 | NA[a] | 100.0 | 107 (15/14) |
| | | | | | | 11,386.0 | 11,373.0 | | 100.0 | |
| | | | | | | 11,403.0 | 11,373.0 | | 100.0 | |
| | | | | | | 11,380.0 | 11,373.0 | | 100.0 | |
| | | | | | | 11,383.0 | 11,373.0 | | 100.0 | |
| | | | | | | 11,342.0 | 11,373.0 | | 99.7 | |
| | | | | | | 11,352.0 | 11,373.0 | | 99.8 | |
| | | | | | | 11,395.0 | 11,373.0 | | 100.0 | |
| | | | | | | 11,349.0 | 11,373.0 | | 99.8 | |
| | | | | | | 11,362.0 | 11,373.0 | | 99.9 | |
| | | | | | | 11,367.0 | 11,373.0 | | 99.9 | |
| Ash | % | 0.40 | 0.31 | 25.4 | % | 2.86 | 2.90 | NA[a] | 98.6 | 64 (9/14) |
| | | | | | | 7.09 | 7.21 | | 98.3 | |
| | | | | | | 7.14 | 7.21 | | 99.0 | |

[a] For spiked samples only.

[b] Excluding QA/QC and blanks.

The completeness goals were exceeded for chlorine and sulfur. The precision achieved on split samples was short of the precision goals for both chlorine and sulfur. For chlorine, no standards were analyzed, although an excess number of spikes were analyzed with good results. For sulfur, no spikes were analyzed, although an excess number of standards were analyzed with excellent results.

All heat value (Btu/lb) goals were met or exceeded.

Completeness goals for ash were not met because waste fuel samples were not analyzed for ash content as planned. The precision result for a split sample was below the expected goal. The analyzed standards exceeded the recovery goals.

## 7.5 QA/QC RESULTS FOR METALS

All samples were digested with hot concentrated nitric acid, diluted to a known volume (usually 50 mL), and then analyzed by ICAP according to EPA Interim Method 200.7. The samples analyzed for metals included the waste fuel, coke, limestone feed, lime product, baghouse dust, and stack gas particulate matter. Fifty-three samples were analyzed.

Five reference standard samples were analyzed for the twenty metals. Results of the percent recovery are summarized in Table 7-7. The average percent recovery is 99.0% ± 4.9% (1 standard deviation), which is within the limits set in Table 7-2; the range is 93% to 130%.

Four samples were split in the lab prior to analyses. Results of the percent recovery ([result of split/result of sample] x 100] also are summarized in Table 7-7. The average percent recovery is 98.9% ± 18.6% (1 standard deviation), which is slightly above the ±15% goal set prior to the test.

In addition, five field splits were made (samples 6A and 6D for each process sample). Results for percent relative difference also are shown in Table 7-7. The average percent relative difference for all metals field splits is 17% ± 30% (1 standard deviation). Almost half of the split results were not quantified because concntrations were below the limit of quantification (BLOQ) for the ICAP instrument.

## TABLE 7-7. QA/QC RESULTS FOR METALS

| Parameter | Standard results: Number of samples | Standard results: Recovery, %: Range | Standard results: Recovery, %: Average | Standard results: Recovery, %: Standard deviation | Split results-lab: Number of samples | Split results-lab: Recovery, %[a]: Range | Split results-lab: Recovery, %[a]: Average | Split results-lab: Recovery, %[a]: Standard deviation | Split results-field: Number of samples[c] | Split results-field: Relative difference, %[b]: Range | Split results-field: Relative difference, %[b]: Average | Split results-field: Relative difference, %[b]: Standard deviation |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Aluminum | 5 | 94-103 | 97.6 | 4.2 | 4 | 62-100 | 85.5 | 16.4 | 3 | 3.6-52 | 22.5 | 26 |
| Boron | 5 | 94-110 | 101 | 7.2 | 4 | BLOQ[d] | - | - | 2 | 0.0-123 | 61.5 | 87 |
| Barium | 3 | 100-102 | 101 | 1.0 | 4 | BLOQ | - | - | 1 | 0.4 | 0.4 | - |
| Beryllium | 5 | 93-100 | 96.4 | 2.6 | 4 | 97-113 | 103 | 7.1 | 3 | 0.5-1.3 | 0.8 | 0.4 |
| Calcium | 0 | - |  | - | 4 | 99-103 | 101 | 1.6 | 3 | 2.3-7.6 | 4.2 | 2.9 |
| Cadmium | 5 | 94-102 | 97.8 | 2.9 | 4 | BLOQ | - | - | 0 | - | - | - |
| Cobalt | 5 | 96-102 | 98.6 | 2.4 | 4 | 88-116 | 103 | 13.4 | 3 | 5.9-26.5 | 13.1 | 11.6 |
| Chromium | 5 | 95-101 | 98.0 | 2.8 | 4 | 91-98 | 95.7 | 3.2 | 4 | 0.0-22.2 | 9.8 | 11.2 |
| Copper | 5 | 97-102 | 98.4 | 2.1 | 4 | 95-103 | 99.3 | 3.3 | 3 | 1.8-6.0 | 3.6 | 2.2 |
| Iron | 5 | 93-130 | 105 | 14.2 | 4 | 92-104 | 97.0 | 6.0 | 5 | 2.1-38.8 | 21.5 | 14.0 |
| Magnesium | 0 | - | - | - | 4 | 99-102 | 102 | 1.3 | 4 | 0.0-173 | 47.4 | 83.7 |
| Manganese | 5 | 97-102 | 98.2 | 2.2 | 4 | 96-102 | 99.8 | 2.9 | 5 | 1.8-24.1 | 10.1 | 8.8 |
| Molybdenum | 5 | 95-108 | 100.8 | 5.8 | 4 | BLOQ | - | - | 0 | - | - | - |
| Sodium | 0 | - | - | - | 4 | 96-103 | 100 | 3.8 | 3 | 1.0-7.9 | 5.2 | 3.7 |
| Nickel | 5 | 95-101 | 97.6 | 2.8 | 4 | 97-101 | 98.5 | 1.9 | 4 | 1.5-38.5 | 14.6 | 16.5 |
| Lead | 5 | 94-103 | 97.2 | 3.4 | 4 | BLOQ | - | - | 2 | 1.6-22.9 | 12.3 | 15.1 |
| Tin | 0 | - | - | - | 4 | BLOQ | - | - | 0 | - | - | - |
| Titanium | 5 | 96-100 | 98.0 | 1.9 | 2 | 27-103[e] | 66.5 | 55 | 2 | 1.4-51.0 | 26.2 | 35.1 |
| Vanadium | 5 | 94-104 | 99.2 | 4.2 | 4 | 94-105 | 101 | 5.3 | 3 | 7.4-8.8 | 8.2 | 0.7 |
| Zinc | 5 | 96-101 | 99.4 | 2.0 | 4 | 58-181 | 119 | 53 | 5 | 1.5-53.3 | 22.9 | 23.1 |

[a] Percent recovery not relative difference used for these samples.

[b] Percent relative difference = $(X_1 - X_2)/\bar{X} \times 100 = (\text{range})/\bar{X} \times 100$.

[c] Number of samples analyzed (five total), excluding the number that were BLOQ.

[d] Below limit of quantification (BLOQ).

[e] Two samples BLOQ.

# APPENDIX A

## Continuous Monitoring Curves for CO, $NO_x$, $SO_2$, $O_2$, and THC

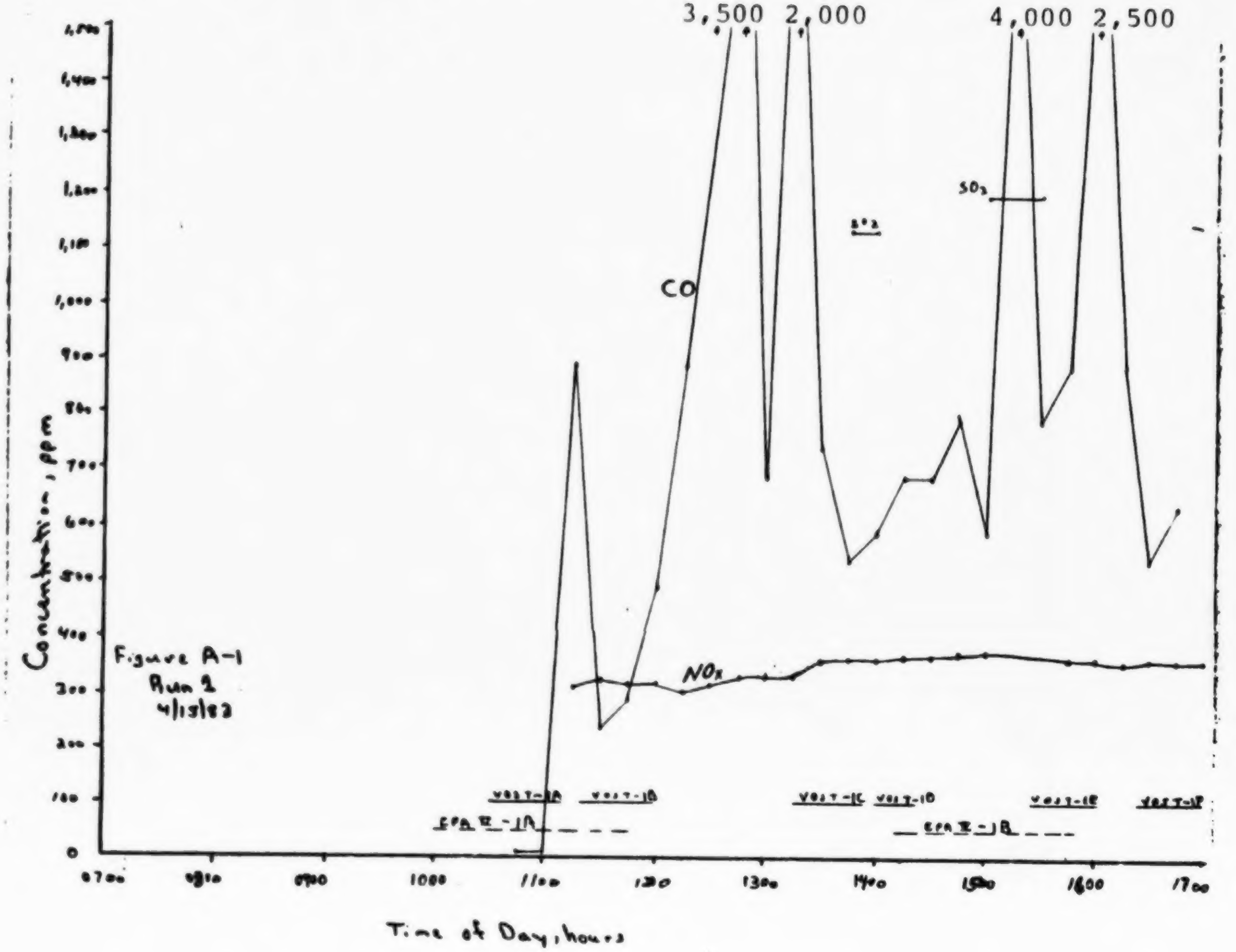

Figure A-1. CO, $NO_x$, $SO_2$ concentrations for Run 1.

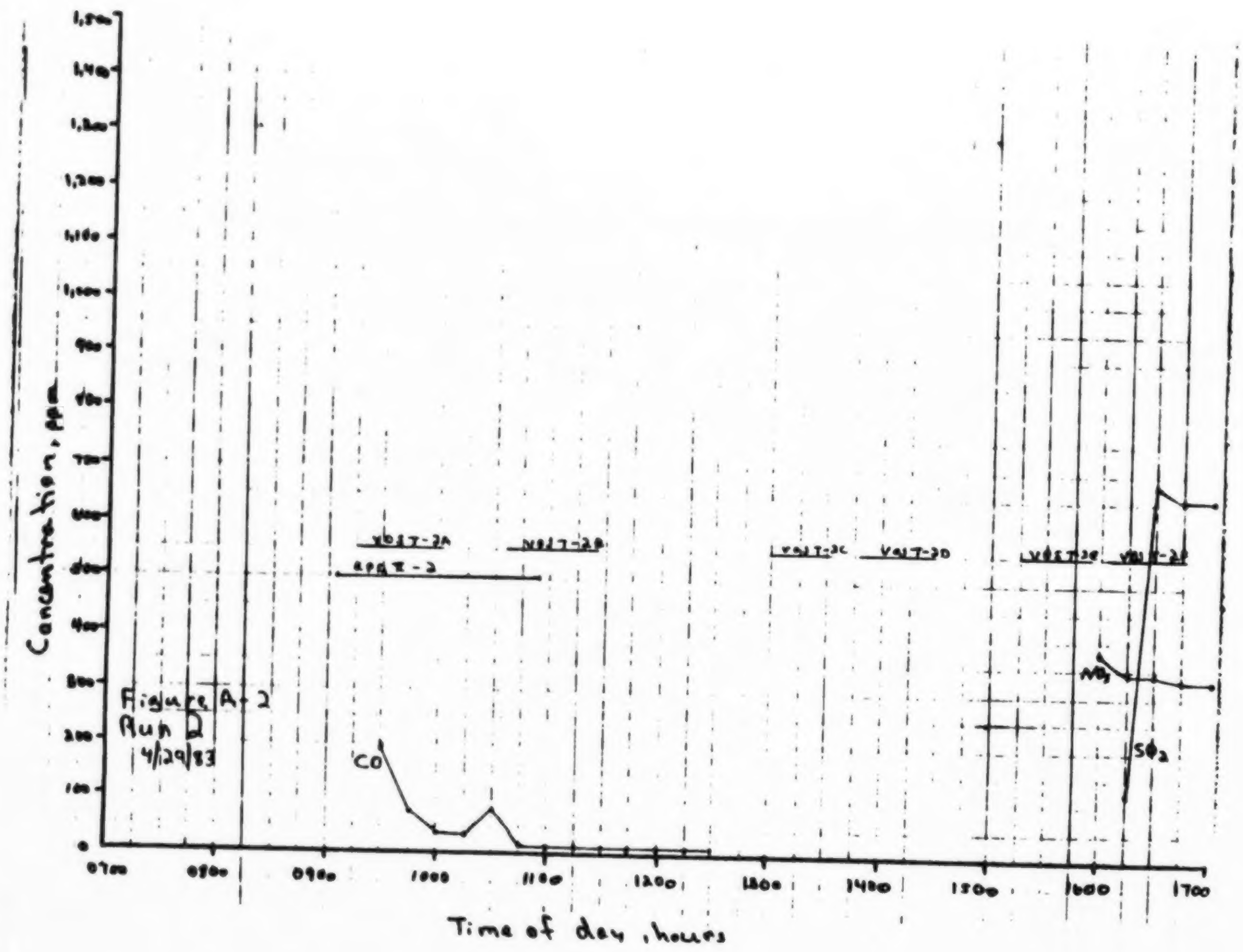

Figure A-2. CO, $NO_x$, $SO_2$ concentrations for Run 2.

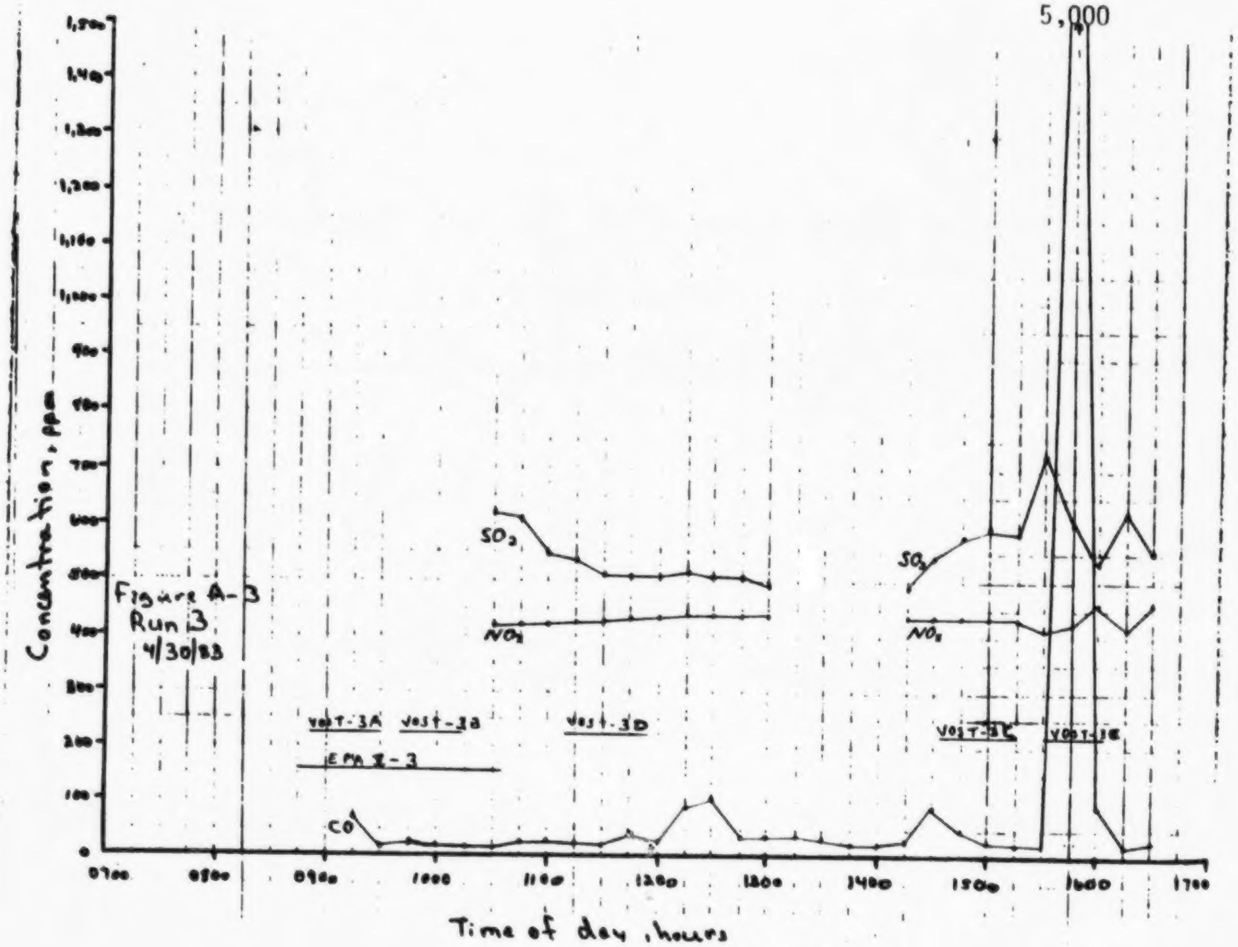

Figure A-3. CO, $NO_X$, $SO_2$ concentrations for Run 3.

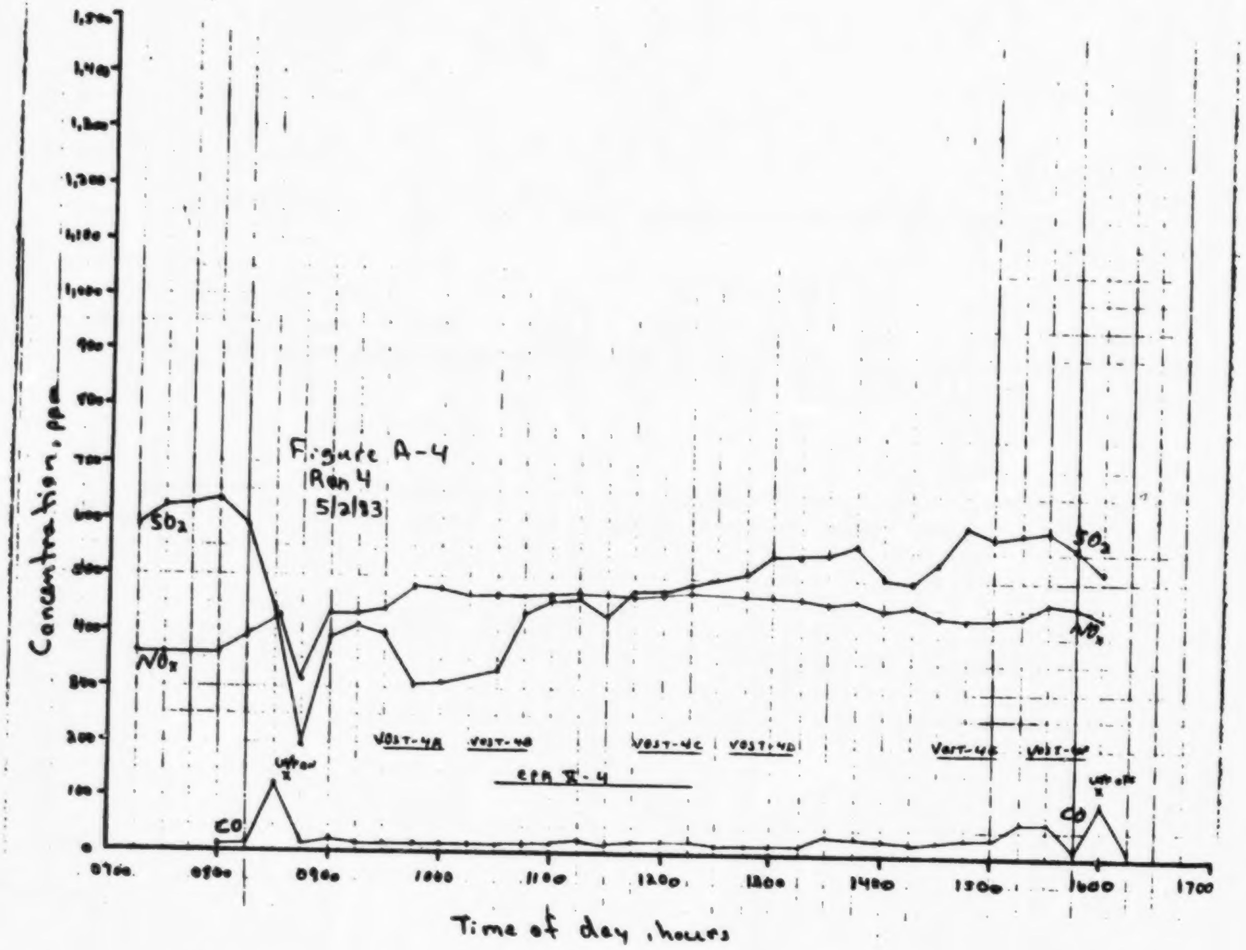

Figure A-4. CO, $NO_X$, $SO_2$ concentrations for Run 4.

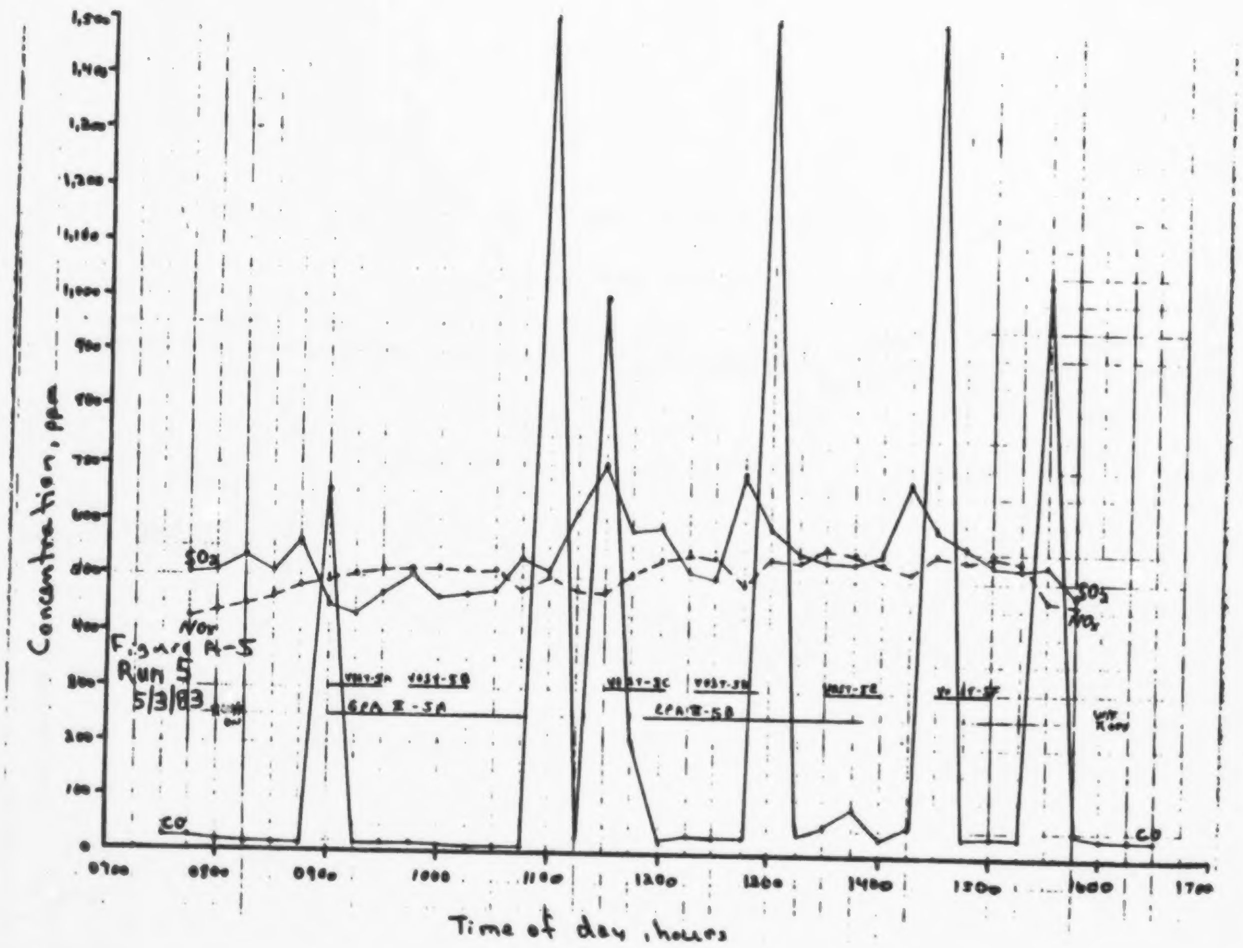

Figure A-5. CO, $NO_x$, $SO_2$ concentrations for Run 5.

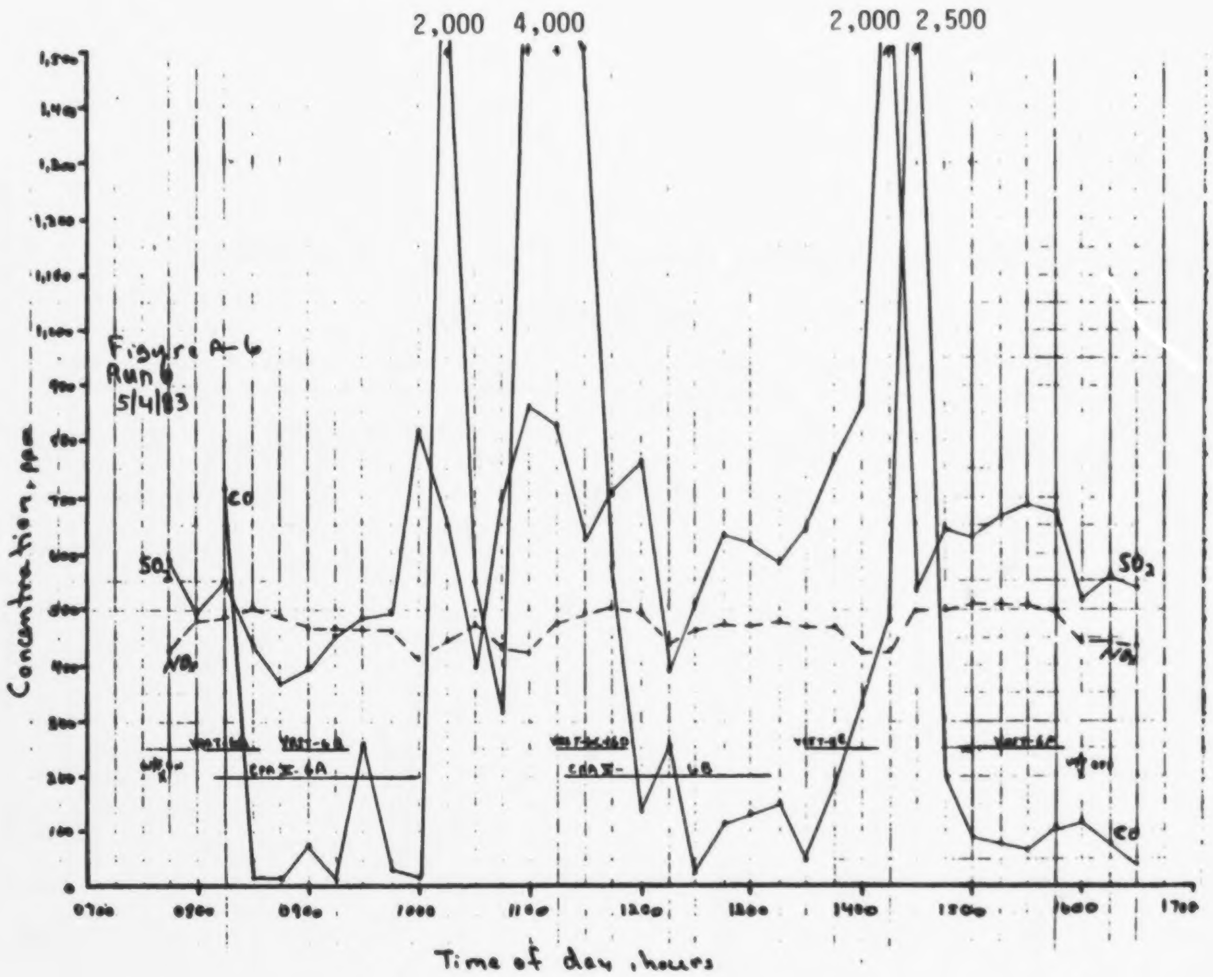

Figure A-6. CO, $NO_x$, $SO_2$ concentrations for Run 6.

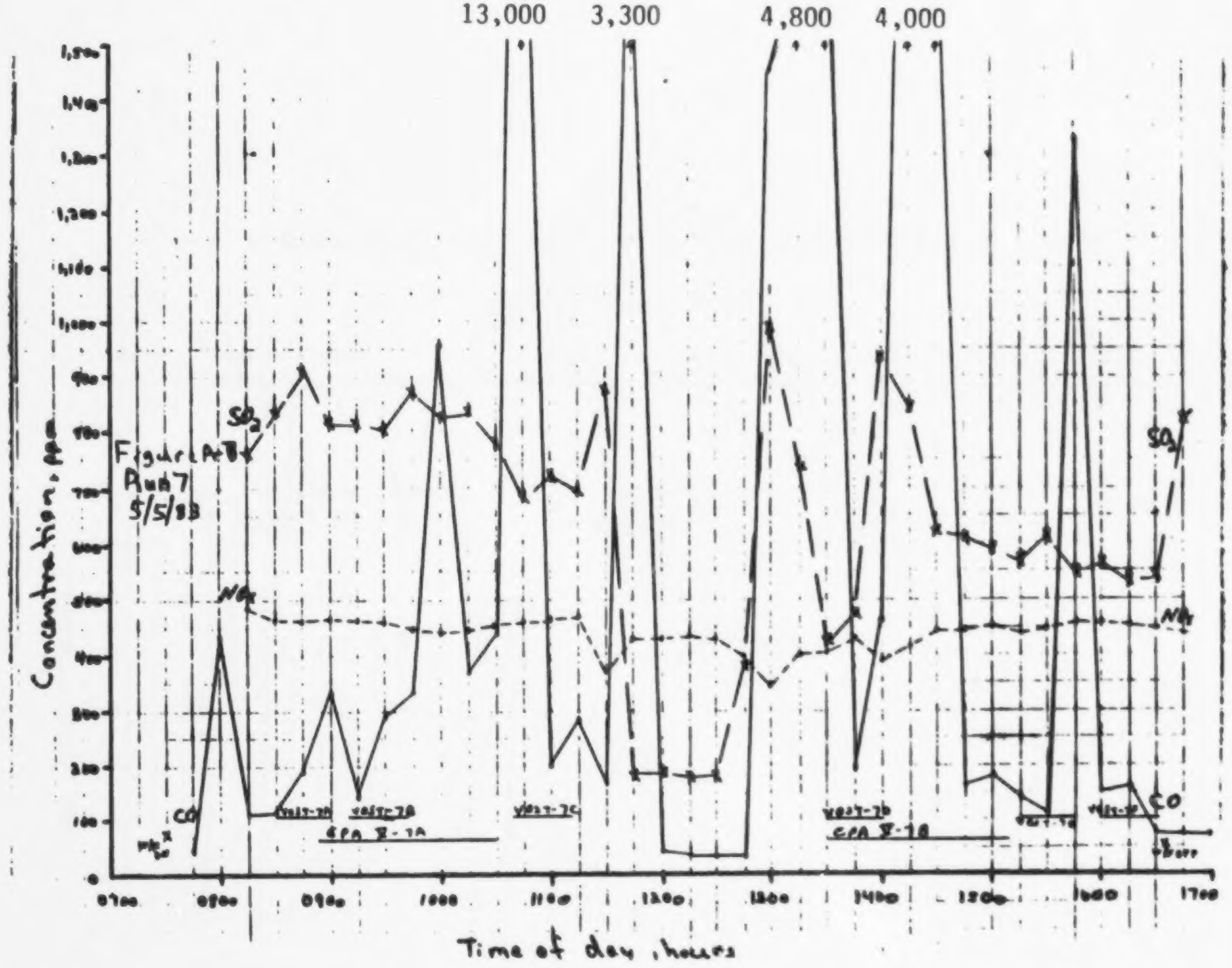

Figure A-7. CO, $NO_x$, $SO_2$ concentrations for Run 7.

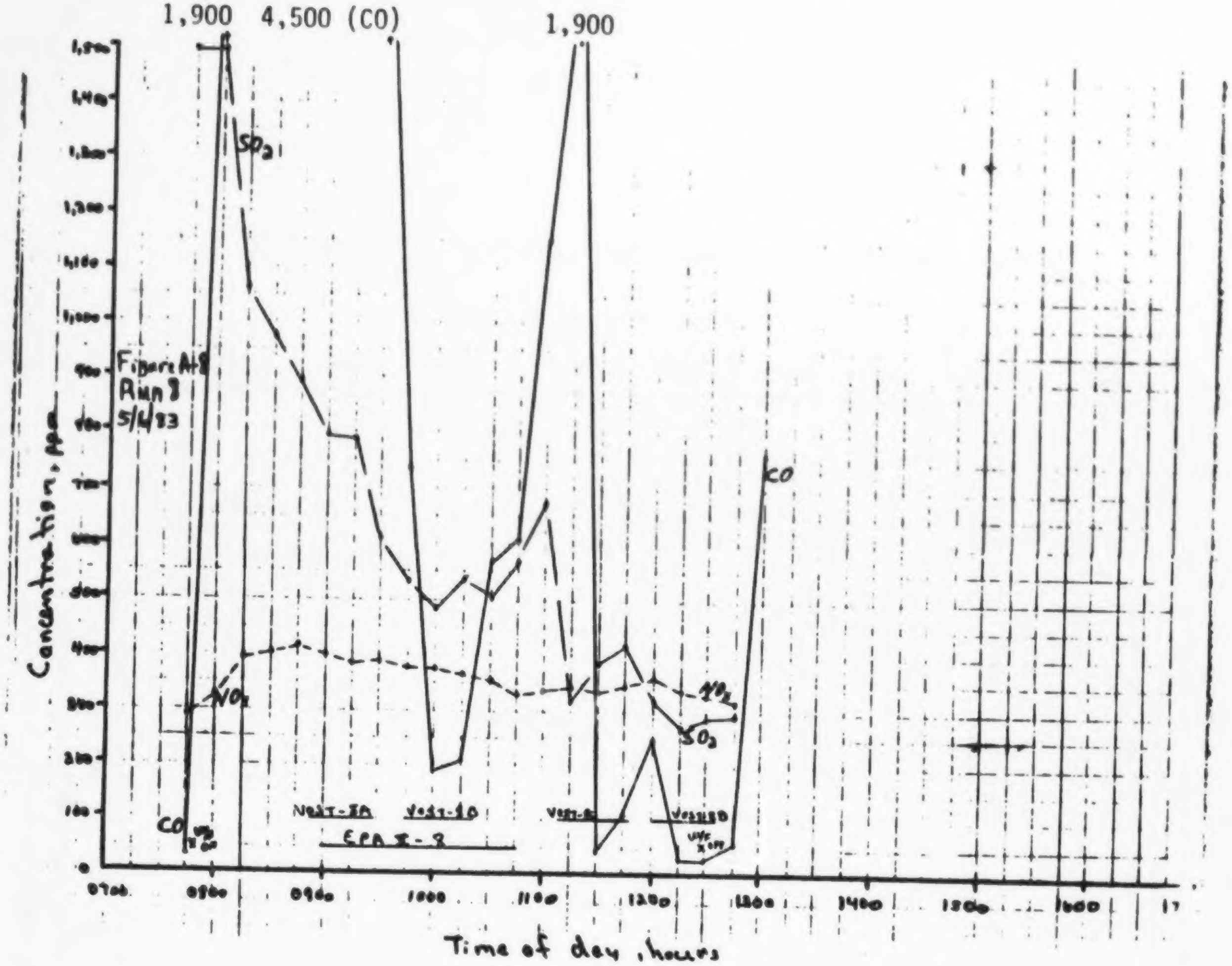

Figure A-8. CO, $NO_x$, $SO_2$ concentrations for Run 8.

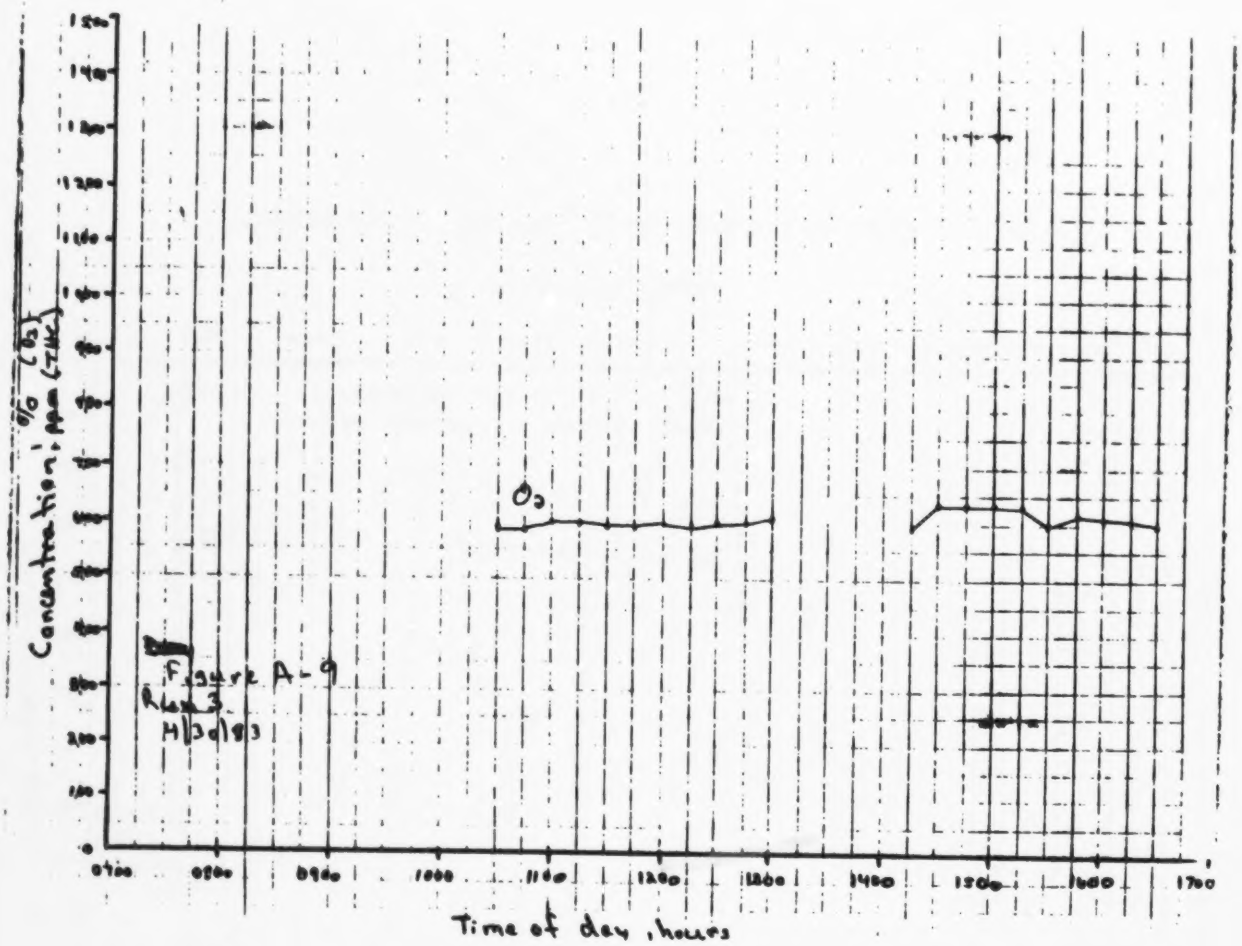

Figure A-9. $O_2$ and THC concentrations for Run 3.

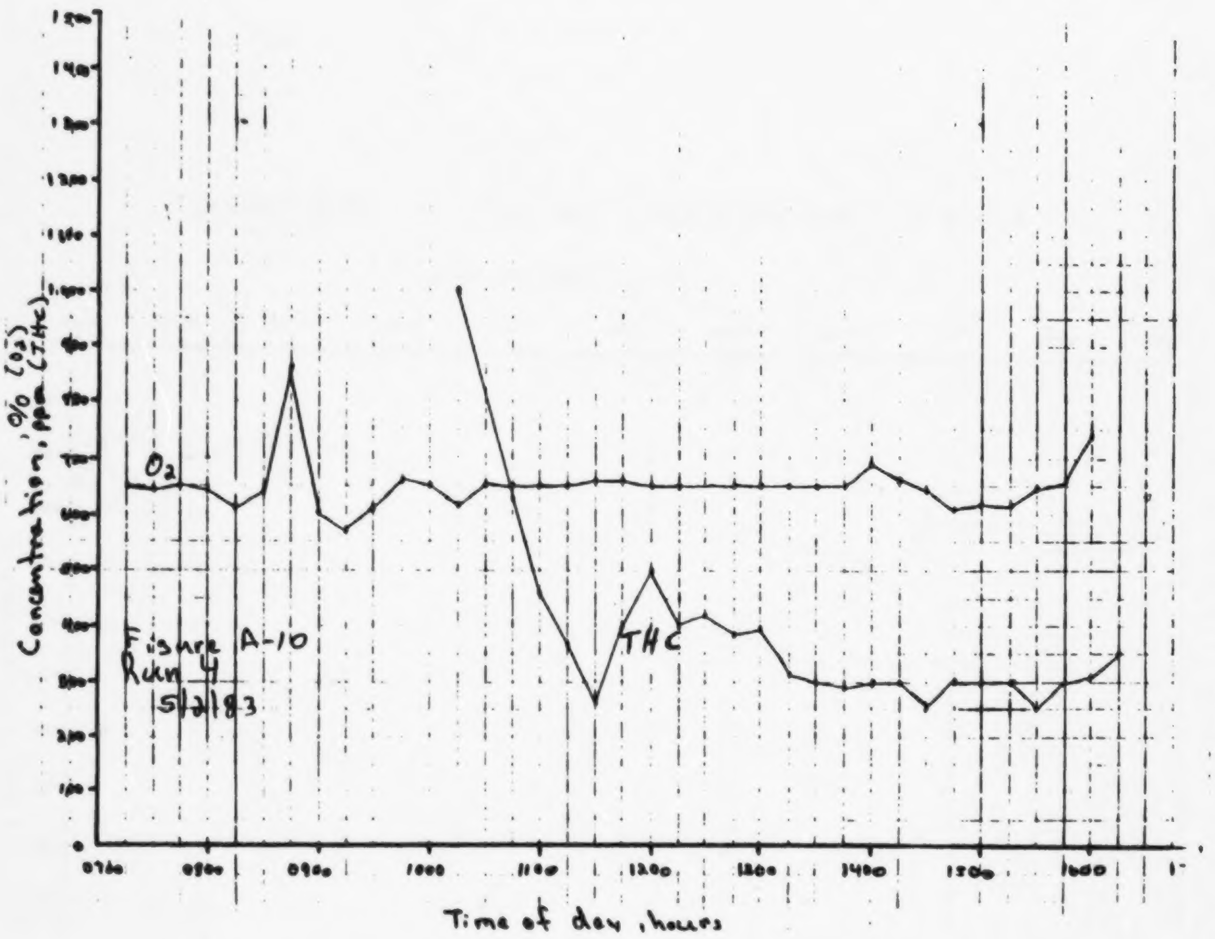

Figure A-10. $O_2$ and THC concentrations for Run 4.

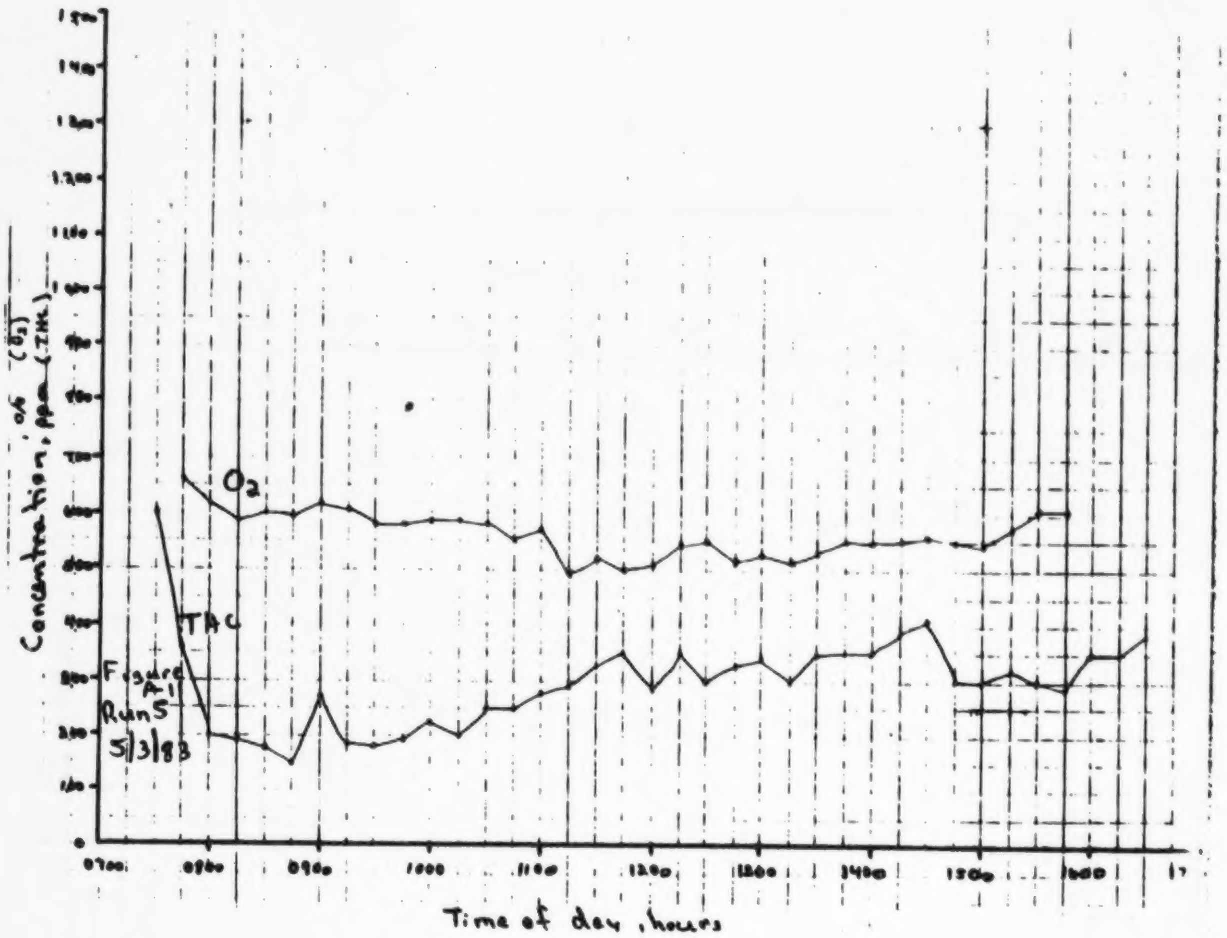

Figure A-11. $O_2$ and THC concentrations for Run 5.

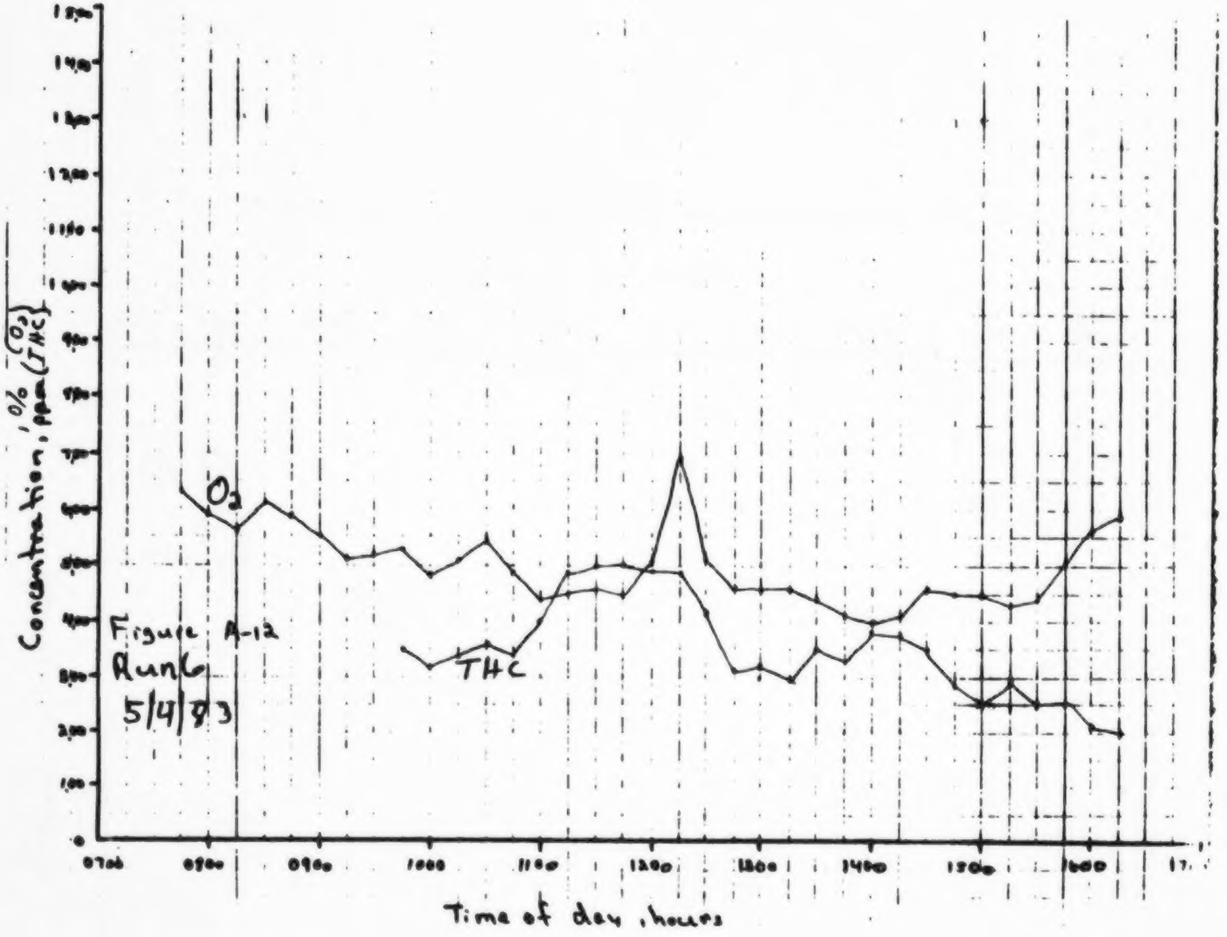

Figure A-12. $O_2$ and THC concentrations for Run 6.

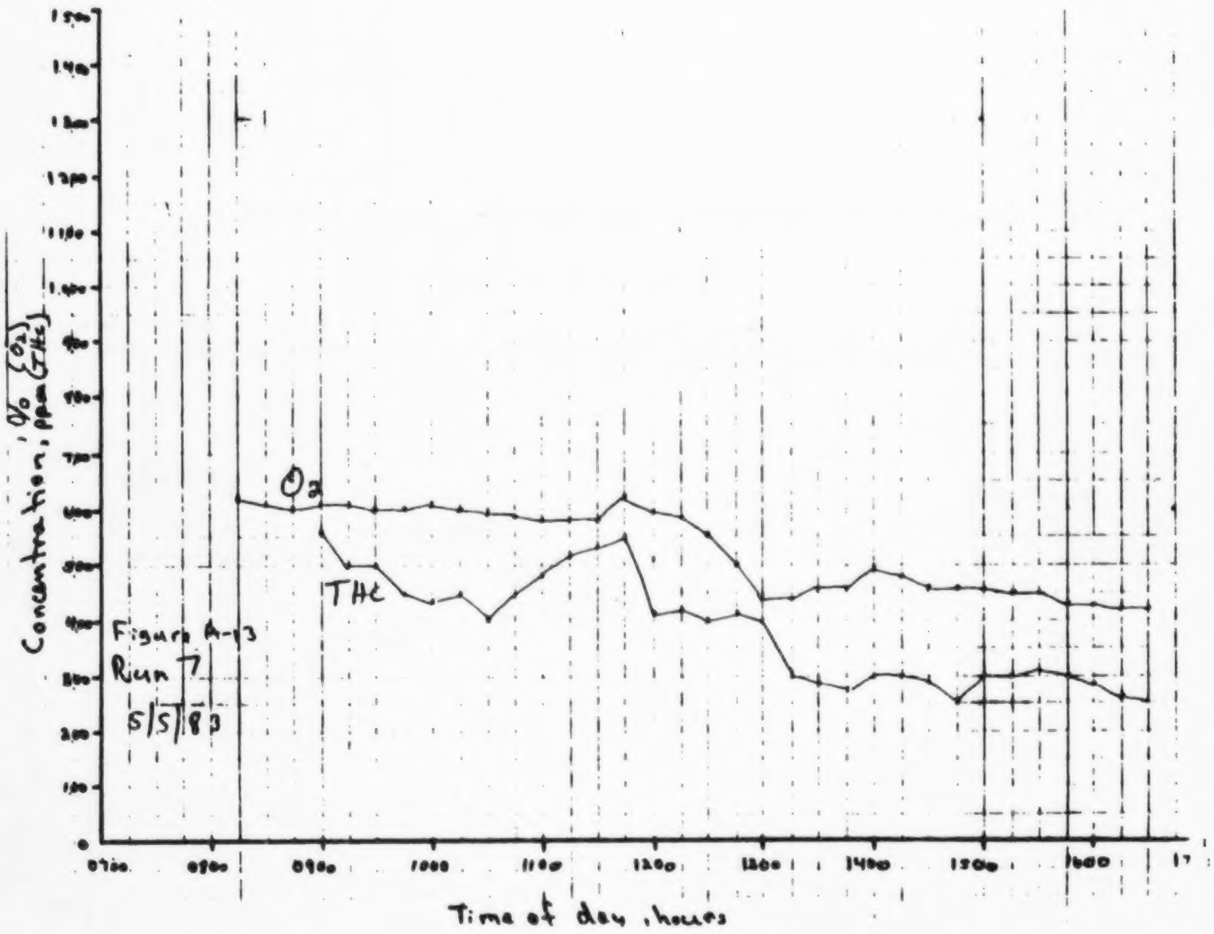

Figure A-13. $O_2$ and THC concentrations for Run 7.

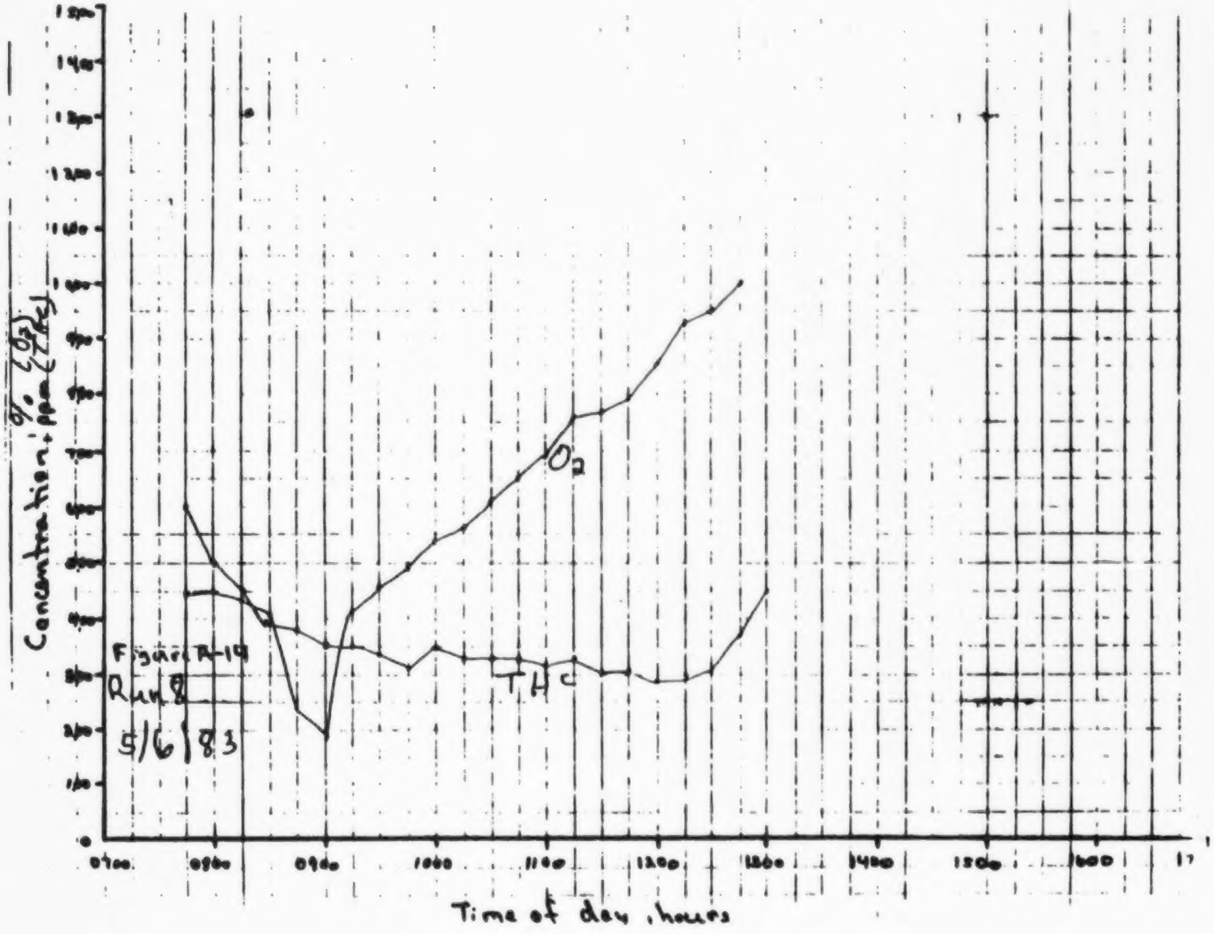

Figure A-14. $O_2$ and THC concentrations for Run 8.

# APPENDIX B

## Analytical Results of Waste Fuels and VOST Tubes

Table B-1 GC/MS Results of Waste Fuels for POHCs

Table B-2 GC/MS Wide Scan Results of Waste Fuels

Table B-3 Thermal Desorption GC/MS Results of VOST Tubes for POHCs

Table B-4 VOST Sample Volumes

## TABLE B-1. RESULTS OF CAPILLARY GC/MS ANALYSIS FOR SIX POHC's IN ROCKWELL WASTE FUELS NO. 4 THROUGH NO. 8

| Compound | Dilution solvent | L.O.D.[a] (wt %) | Solvent blank | Waste fuel No. 4 | Waste fuel No. 4 dup. | % Relative difference | Waste fuel No. 5 | Waste fuel No. 5 dup. | % Relative difference | Waste fuel No. 6A | Waste fuel No. 6A dup. | Waste fuel No. 6B | Waste fuel No. 6B dup. | % Relative difference |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Methylene chloride | Nonane | 0.05 | ND[b] | 0.108 | 0.093 | 14.9 | 0.088 | 0.105 | 17.6 | 0.115 | 0.099 | 0.108 | 0.103 | 15.1 |
| Methyl ethyl ketone | Nonane | 0.01 | ND | 2.48 | 2.47 | 0.4 | 3.04 | 2.46 | 21.1 | 2.55 | 2.56 | 2.16 | 2.64 | 19.4 |
| 1,1,1,-Trichloroethane | Nonane | 0.01 | ND | 0.255 | 0.221 | 14.3 | 0.250 | 0.227 | 9.6 | 0.216 | 0.213 | 0.233 | 0.250 | 16.2 |
| Trichloroethylene | Nonane | 0.01 | ND | 1.97 | 1.49 | 27.8 | 1.78 | 1.49 | 17.7 | 1.80 | 1.50 | 1.85 | 1.95 | 25.4 |
| Tetrachloroethylene | Nonane | 0.01 | ND | 2.26 | 2.08 | 8.3 | 2.09 | 1.95 | 6.9 | 2.14 | 2.14 | 1.94 | 1.96 | 9.8 |
| Toluene | Nonane | 0.005 | ND | 12.0 | 9.94 | 18.8 | 10.9 | 10.2 | 6.6 | 11.5 | 11.1 | 10.4 | 10.8 | 10.0 |

(continued)

## TABLE B-2. RESULTS OF CAPILLARY GC/MS ANALYSIS FOR MAJOR COMPONENTS IN ROCKWELL WASTE FUELS NO. 4 AND NO. 7

| | | | Concentration (wt %) | | | | | |
|---|---|---|---|---|---|---|---|---|
| Waste Fuel Components | Quantifying compound | Quantifying Ion | Waste fuel No. 4 | Waste fuel No. 4 (duplicate) | % Relative difference | Waste fuel No. 7 | Waste fuel No. 7 (duplicate) | % Relative difference |
| Acetone | Acetone | TI[a] | 0.242 | 0.216 | 11.4 | 0.228 | 0.218 | 4.5 |
| Methyl ethyl ketone | Methyl ethyl ketone | TI | 2.48 | 2.47 | 0.4 | 2.99 | 3.34 | 5.5 |
| 1,1,1-Trichloroethane | 1,1,1-Trichloroethane | TI | 0.255 | 0.221 | 14.3 | 0.198 | 0.233 | 16.2 |
| 1-Butanol | 1-Butanol | 56 | 0.325 | 0.316 | 2.8 | 0.350 | 0.390 | 10.8 |
| Trichloroethylene | Trichloroethylene | TI | 1.97 | 1.49 | 27.7 | 2.38 | 1.93 | 20.9 |
| 2-Ethoxyethanol | 2-Ethoxyethanol | TI | 0.872 | 0.831 | 4.8 | 0.890 | 0.940 | 8.1 |
| Methyl isobutyl ketone | Methyl isobutyl ketone | TI | 1.14 | 0.982 | 13.8 | 1.13 | 1.20 | 6.0 |
| Toluene | Toluene | TI | 12.0 | 9.94 | 18.8 | 12.6 | 12.4 | 1.6 |
| Tetrachloroethylene | Tetrachloroethylene | TI | 2.26 | 2.08 | 8.7 | 2.45 | 2.52 | 15.8 |
| Butyl acetate | Methyl isobutyl ketone | TI | 0.281 | 0.261 | 7.4 | 0.285 | 0.350 | 20.5 |
| Ethylbenzene | Ethylbenzene | TI | 1.44 | 1.39 | 3.5 | 1.65 | 1.51 | 8.9 |
| Xylene (isomer #1) | O-Xylene | TI | 4.96 | 4.88 | 1.6 | 5.73 | 5.42 | 5.6 |
| Xylene (isomer #2) | O-Xylene | TI | 1.46 | 1.40 | 4.2 | 1.64 | 1.56 | 5.0 |
| 2-Butoxyethanol | 2-Ethoxyethyl acetate | TI | 2.06 | 1.92 | 7.0 | 2.11 | 2.03 | 3.9 |
| 2-Ethoxyethyl acetate | 2-Ethoxyethyl acetate | TI | 6.07 | 5.74 | 5.6 | 6.56 | 6.18 | 6.0 |
| $C_3$-Benzene (isomer #1) | 1,2,4-Trimethylbenzene | TI | 0.318 | 0.250 | 23.9 | 0.345 | 0.299 | 14.3 |
| $C_3$-Benzene (isomer #2) | 1,2,4-Trimethylbenzene | TI | 0.458 | 0.470 | 25.9 | 0.565 | 0.579 | 2.4 |
| $C_{10}$-Alkane | Decane | TI | 0.831 | 0.770 | 7.6 | 0.966 | 0.922 | 4.8 |
| Alkane-$C_8$ | Decane | TI | 0.228 | 0.253 | 10.4 | 0.280 | 0.273 | 2.5 |
| Alkane | Decane | TI | 0.155 | 0.134 | 14.5 | 0.183 | 0.174 | 4.5 |
| $C_{11}$-Alkane | Undecane | TI | 1.29 | 1.23 | 4.8 | 1.51 | 1.45 | 10.1 |
| 2-Cyclohexen-1-one 3,5,5-Trimethyl (or isomer) | Undecane | TI | 0.160 | 0.151 | 5.8 | 0.161 | 0.197 | 20.1 |
| Alkane-$C_6$ | Dodecane | TI | 0.211 | 0.196 | 7.4 | 0.246 | 0.240 | 1.2 |

[a] TI = Total ion.

## TABLE B-3. RESULTS OF THERMAL DESORPTION GC/MS ANALYSIS OF ROCKWELL VOST TUBES FOR SIX POHC's

| | Total ng on Tubes | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Compound | Lab blank | Lab blank | Lab blank | Lab blank | Trip blank | Trip blank | 1 A-F | 1 A-B | 1 B-F | 1 B-B | 1 C-F | 1 C-B | 1 D-F | 1 D-B |
| Methylene chloride[a] | 3.85 | 1.85 | 0.71 | 4.85 | 0.46 | 0.97 | 0.58 | 1.33 | 1.63 | NAI[b] | 1.21 | 0.61 | 0.65 | 0.67 |
| Methyl ethyl ketone | 5.34 | 1.58 | 4.96 | 10.4 | 2.40 | 2.78 | 18.0 | 5.54 | 3.57 | NAI | 4.23 | 9.25 | 5.47 | 4.63 |
| 1,1,1-Trichloroethane | 55.6 | 12.7 | 3.46 | 8.01 | 1.77 | 8.08 | 3.88 | 4.43 | 6.70 | NAI | 1.94 | 84.1 | 5.37 | 6.15 |
| Trichloroethylene | 2.60 | 7.31 | 0.98 | 4.53 | 1.25 | 2.46 | 191.5 | 9.0 | 5.89 | NAI | 4.26 | 2.03 | 2.75 | 1.85 |
| Tetrachloroethylene | 1.72 | 1.45 | 2.70 | 5.32 | 1.22 | 5.40 | 4.50 | NAI | 3.59 | NAI | NAI | 7.81 | 5.89 | 5.10 |
| Toluene | 6.05 | 18.1 | 4.59 | 12.7 | 2.50 | 10.7 | 90.3 | NAI | 47.0 | NAI | NAI | 19.4 | 78.9 | 23.9 |

(continued)

## TABLE B-3 (continued)

| Compound | Total ng on Tubes<br>8 A-F | 8 A-B | 8 B-F | 8 B-B | 8 C-F | 8 C-B | 8 D-F | 8 D-B | 8 E-F Field blank | 8 E-B Field blank | 8 F-F Field blank | 8 F-B Field blank |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Methylene chloride | NAI | 6.16 | 8.56 | 3.11 | 0.81 | 3.99 | 1.59 | 1.35 | 0.62 | 5.31 | 0.43 | 0.31 |
| Methyl ethyl ketone | NAI | 15.6 | 45.8 | 12.4 | ND | 17.5 | 66.8 | 30.8 | 4.56 | 6.59 | 3.30 | 2.47 |
| 1,1,1-Trichloroethane | NAI | 22.3 | 29.9 | 10.8 | 26.6 | 13.5 | 16.0 | 65.6 | 3.80 | 25.9 | 2.62 | 4.41 |
| Trichloroethylene | 11.3 | 6.55 | 11.0 | 3.59 | 23.7 | 4.07 | 9.01 | 31.6 | 0.92 | 17.6 | 1.96 | 2.41 |
| Tetrachloroethylene | 14.6 | 14.9 | 33.1 | 11.5 | 59.3 | 7.09 | 22.2 | 18.6 | 6.54 | 10.5 | 4.59 | 5.33 |
| Toluene | 238.2 | 34.2 | 139.2 | 15.5 | 2.03 | 14.3 | 122.9 | 44.1 | 5.95 | 21.2 | 6.39 | 7.29 |

[a] Data presented for methylene chloride are questionable since methylene chloride extracts of samples were routinely analyzed in the same laboratory as were the Tenax tubes. The day-to-day variation in response factors for methylene chloride was much greater than for the other analytes, suggesting that contamination of the standard tube may have been a problem. Sample tubes may have likewise been contaminated.

[b] NAI = Not reported because of instrument problem, i.e., software problem or power outage.

[c] NAT = Not analyzed because ends of tubes were broken off too short.

[d] NAS = Not analyzed because sample was not received.

[e] NAP = Not analyzed because tube was plugged, possibly due to large amounts of water vapor.

[f] ND = Not detected, with a detection limit of 0.5 ng.

## TABLE B-4 VOST SAMPLE VOLUMES AT ROCKWELL LIME

| | Sampling Conditions[a] | | | $V_{m\ std.}$ | |
|---|---|---|---|---|---|
| Run No. | Sample volume, $V_m$ ($ft^3$) | Temperature, $T_m$ (°F) | Pressure, $P_{bar}$ (in Hg) | dscf[b] | dscm[c] |
| 1A | 1.060 | 64.6 | 29.08 | 1.0330 | 0.0292 |
| 1B | 1.060 | 72.9 | 29.08 | 1.0167 | 0.0288 |
| 1C | 1.060 | 70.0 | 29.08 | 1.0222 | 0.0290 |
| 1D | 1.060 | 74.3 | 29.08 | 1.0140 | 0.0287 |
| 1E | 1.064 | 73.0 | 29.08 | 1.0203 | 0.0289 |
| 1F | 1.064 | 76.0 | 29.10 | 1.0153 | 0.0288 |
| 1G | Field Blank | | | | |
| 1H | Field Blank | | | | |
| 1I | Trip Blank | | | | |
| 2A | 1.296 | 61.1 | 29.15 | 1.2742 | 0.0361 |
| 2B | 1.206 | 72.8 | 29.17 | 1.1605 | 0.0329 |
| 2C | 1.082 | 70.0 | 29.17 | 1.0467 | 0.0296 |
| 2D | 1.104 | 77.5 | 29.17 | 1.0530 | 0.0298 |
| 2E | 1.068 | 80.0 | 29.17 | 1.0140 | 0.0287 |
| 2F | 1.094 | 81.7 | 39.17 | 1.0354 | 0.0293 |
| 3A | 1.128 | 69.8 | 29.08 | 1.0882 | 0.0308 |
| 3B | 1.070 | 69.3 | 29.08 | 1.0332 | 0.0293 |
| 3C | 1.088 | 74.0 | 29.08 | 1.0414 | 0.0295 |
| 3D | 1.176 | 68.0 | 29.08 | 1.1384 | 0.0322 |
| 3E | 0.975 | 80.3 | 29.14 | 0.9223 | 0.0261 |
| 4A | 1.121 | 60.0 | 28.50 | 1.0799 | 0.0306 |
| 4B | 1.175 | 76.0 | 28.50 | 1.0981 | 0.0311 |
| 4C | 1.035 | 78.0 | 28.50 | 0.9637 | 0.0273 |
| 4D | 1.078 | 79.0 | 28.57 | 1.0043 | 0.0284 |
| 4E | 1.013 | 80.0 | 28.65 | 0.9446 | 0.0268 |
| 4F | 1.105 | 80.0 | 28.65 | 1.0304 | 0.0292 |
| 5A | 0.947 | 68.0 | 28.86 | 0.9098 | 0.0258 |
| 5B | 1.034 | 76.0 | 28.86 | 0.9785 | 0.0277 |
| 5C | 1.340 | 80.0 | 28.86 | 1.2587 | 0.0356 |
| 5D | 1.150 | 86.0 | 28.86 | 1.0684 | 0.0303 |
| 5E | 1.079 | 84.0 | 28.86 | 1.0061 | 0.0285 |
| 5F | 1.182 | 88.0 | 28.86 | 1,0941 | 0.0310 |

(continued)

TABLE B-4 (continued)

| | Sampling Conditions[a] | | | $V_{m\ std.}$ | |
|---|---|---|---|---|---|
| Run No. | Sample volume, $V_m$ ($ft^3$) | Temperature, $T_m$ (°F) | Pressure, $P_{bar}$ (in Hg) | dscf[b] | dscm[c] |
| 6A | 1.267 | 63.7 | 28.75 | 1.2225 | 0.0346 |
| 6B | 1.065 | 72.9 | 28.75 | 1.0099 | 0.0286 |
| 6C | 1.205 | 83.6 | 28.75 | 1.1201 | 0.0317 |
| 6D | 1.292 | 65.9 | 28.75 | 1.2414 | 0.0352 |
| 6E | 0.981 | 83.6 | 28.86 | 0.9154 | 0.0259 |
| 6F | 1.004 | 82.0 | 28.86 | 0.9396 | 0.0266 |
| 7A | 1.062 | 62.6 | 29.19 | 1.0426 | 0.0295 |
| 7B | 0.785 | 72.8 | 29.19 | 0.7559 | 0.0214 |
| 7C | 0.999 | 79.6 | 29.19 | 0.9498 | 0.0269 |
| 7D | 1.059 | 70.0 | 29.19 | 1.0251 | 0.0290 |
| 7E | 1.007 | 79.0 | 29.19 | 0.9585 | 0.0271 |
| 7F | 0.714 | 79.8 | 29.19 | 0.6786 | 0.0192 |
| 8A | 0.964 | 63.1 | 29.07 | 0.9416 | 0.0267 |
| 8B | 1.017 | 73.6 | 29.07 | 0.9738 | 0.0276 |
| 8C | 1.140 | 78.0 | 29.07 | 1.0826 | 0.0307 |
| 8D | 0.898 | 79.0 | 29.07 | 0.8323 | 0.0236 |
| 8E | Field Blank | | | | |
| 8F | Field Blank | | | | |

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[a] From VOST data sheets.

[b] $V_{m\ std.} = V_m\ Y\ \frac{T_{std}}{T_m}\ \frac{P_{bar}}{P_{std.}}$ where Y = 0.996, $T_{std.}$ = 528°F, $P_{std.}$ = 29.92 in Hg (except run 6D Y = 1.019)

[c] 1 $m^3$ = 35.31 $ft^3$